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Luminescence Dating

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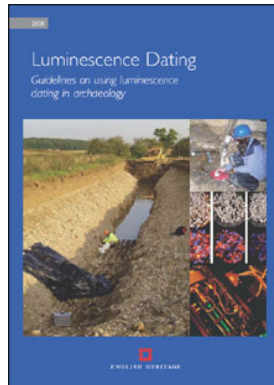
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Luminescence Dating



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2008

Luminescence Dating

Guidelines on using luminescence dating in archaeology



ENGLISH HERITAGE

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Preface

These guidelines are designed to establish good practice in the use of luminescence dating for providing chronological frameworks. They provide practical advice on using luminescence dating methods in archaeology. The guidelines should not be regarded as a substitute for advice given by specialists on specific projects; and, given how rapidly the methods have developed, it is likely that further improvements in laboratory techniques will occur, so more up to date advice is likely to become available in the future.

The guidelines will help archaeologists and site investigators to assess whether luminescence dating will be of value in providing chronological information for understanding their site. They are divided into three main parts: Part A, an introduction to luminescence dating, including the principles underlying the method and the measurement procedures used; Part B, a section on the practicalities of collecting samples, collaborating with a luminescence laboratory, understanding the results obtained and presenting luminescence ages; and Part C, a series of case studies to illustrate the use of the method.

The first section is designed to enable the non-specialist to understand the physical principles underlying luminescence dating so that he or she is more fully able to understand the issues that may affect the reliability of luminescence ages, and critically assess the results from luminescence laboratories.

What these guidelines cover

- an introduction to luminescence dating
- a summary of the variety of luminescence methods available
- a description of the options available for estimating the radiation dose rate at a site
- practical advice about the collection of samples
- a summary of the information that should be provided by laboratories undertaking luminescence measurements, and about how luminescence ages should be quoted
- case studies illustrating different applications of luminescence dating, and the results that can be obtained

How to use these guidelines

Luminescence dating is a technical topic involving consideration of a number of complex scientific issues. These guidelines have presented these topics as clearly as possible, but inevitably there may be some terms and abbreviations that are not familiar to all readers. A list of abbreviations is given in Section 1.1 and a glossary of terms is provided

at the end of these guidelines to help the reader; and text in bold indicates that the term is defined in the glossary. Under Further Reading is a range of other texts to augment the information provided here.

In these guidelines, section 1 provides a summary of the method and section 5 gives some indications of what samples are suitable and what age limits are appropriate. The remainder of Part A contains a more detailed explanation of the techniques involved in the various measurements that are required to obtain a luminescence age. A minimum reading of Part A would be sections 1 and 5. The detailed information provided in sections 2, 3 and 4 is necessary to enable users of luminescence dating to be able to assess what is feasible when using it, and to interpret critically the results that are obtained from a luminescence laboratory.

Part B includes a range of practical considerations, including sample selection and collection. It defines the information that should be required in reports obtained from luminescence laboratories, explains how luminescence ages should be turned into dates and how they should be quoted in site reports or other publications. Some examples of how the method can be applied are given in Part C. An Executive Summary concludes the guidelines.

Part A Introduction to luminescence

Luminescence dating is a chronological method that has been used extensively in archaeology and the earth sciences. It is based on the emission of light, luminescence, by commonly occurring minerals, principally quartz. The method can be applied to a wide range of materials that contain quartz or similar minerals. For pottery, burnt flints and burnt stones, the event being dated is the last heating of the objects. Another, and now very common, application is to date sediments, and in this case the event being dated is the last exposure of the mineral grains to daylight (Fig 1). The age range over which the method can be applied is from a century or less to over one hundred thousand years.

Some of the first applications of luminescence dating were developed in the 1960s. Since that time there has been enormous progress in understanding of the luminescence phenomena in natural minerals, of the methods used to measure that luminescence and in the range of materials that are analysed. To the non-specialist, luminescence can be complex and the results

difficult to interpret. This document is designed to help users employ the available techniques effectively on their projects, and to interpret the results they can obtain from luminescence laboratories.

I An overview of luminescence dating

Radioactivity is ubiquitous in the natural environment. Luminescence dating exploits the presence of radioactive **isotopes** of elements such as uranium (U), thorium (Th) and potassium (K). Naturally occurring minerals such as quartz and feldspars act as dosimeters, recording the amount of radiation to which they have been exposed.

A common property of some naturally occurring minerals is that when they are exposed to emissions released by radioactive decay, they are able to store within their crystal structure a small proportion of the energy delivered by the radiation. This energy accumulates as exposure to radioactive decay continues through time. At some later date this energy may be released, and in some minerals this energy is released in the form of light. This

light is termed luminescence.

What makes this a useful phenomenon for dating? The answer lies in the fact that this energy stored in minerals can be reset by two processes. The first is by heating the sample to temperatures above about 300°C, as would occur in a hearth, or in a kiln during firing of pottery. The second process is exposure of the minerals to daylight, as may occur during erosion, transport and deposition of sediments. Either of these processes will release any pre-existing energy stored, and thus set the 'clock' to zero. Thus in luminescence dating, the event being dated is this resetting, either by heat or by exposure to light.

Measurements of the brightness of the luminescence signal can be used to calculate the total amount of radiation to which the sample was exposed during the period of burial. If this is divided by the amount of radiation that the sample receives from its surroundings each year then this will give the duration of time that the sample has been receiving energy.

$$\text{age} = \frac{\text{total energy accumulated during burial}}{\text{energy delivered each year from radioactive decay}}$$



Fig 1 Excavation at Gwithian, Cornwall in 2005, showing the collection of samples of sands for luminescence dating (© Historic Environment Service, Cornwall County Council 2005).

The SI (Système International) unit of absorbed radiation is the **Gray (Gy)**. It is a measure of the amount of energy absorbed by a sample, or its dose, and has the units joules per kilogramme (J.kg⁻¹). Laboratory luminescence measurements are used to calculate the total absorbed dose. The name given to the quantity is the **equivalent dose (D_e)**. The amount of energy absorbed per year from radiation in the environment surrounding the measured material (known as the **dose rate**) can either be derived by directly measuring the amount of radioactivity, or by chemically analysing the surrounding material and calculating the concentration of radioactive isotopes in it; this has the units of Gray per year. Thus the age equation for luminescence can formally be expressed as:

$$\text{age (years)} = \frac{\text{equivalent dose (D}_e\text{) (Gy)}}{\text{dose rate (Gy/year)}}$$

To understand how this process can be used for dating, a useful analogy is that of a rechargeable battery, in which the battery represents the mineral grains (Fig 2). Exposing mineral grains to light or heat will release the battery's energy, so that when the mineral (battery) is incorporated into sediment or fired in a brick or sherd of pottery, it has no energy. At this time the battery starts to be recharged by exposure to radiation in its natural environment. As time progresses, the stored energy increases. When the sample is collected and measured in the laboratory this releases the energy and light is created. This is

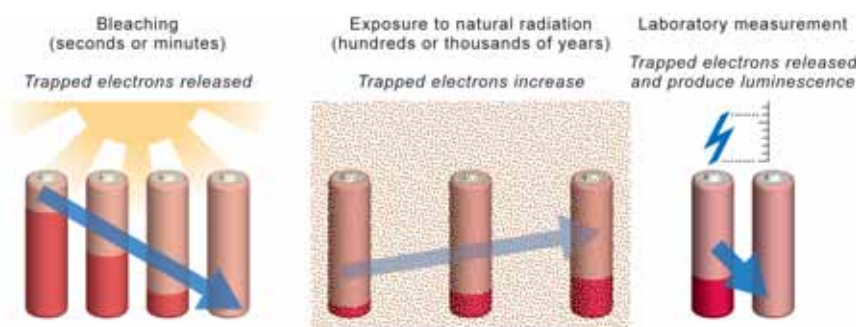


Fig 2 A rechargeable battery provides a useful analogy for how mineral grains behave and how luminescence dating works. When mineral grains are exposed to light or heated, trapped electrons are released, much like depleting a battery. Once this heating or exposure to daylight stops, re-exposure to natural radioactivity begins to recharge the mineral grains, much like recharging a battery. In the laboratory the mineral grains are stimulated to release their stored energy in the form of light emission. The brightness of this luminescence signal can be related to the amount of energy that has been stored in the mineral.

the luminescence signal that is observed. The brightness of this luminescence signal is related to the amount of energy in the battery. If we also know the rate at which the battery was recharged then we can work out how long it must have been recharging, thus telling us the period of time that has elapsed since the battery was last emptied.

Many naturally occurring minerals will yield luminescence signals, including quartz, feldspars, calcite and zircons. Quartz is the most suitable material for dating, and the **single aliquot regenerative dose (SAR)** protocol is now used routinely. The luminescence age is the period of time that has elapsed since the sample was heated (in the case of pottery, burnt flints or bricks) or exposed to daylight (in the case of sediments). The age is given as the number of years before the date of measurement, and the unit of time used is the annum (abbreviated 'a'). There is no convention for the datum to which luminescence ages are referred, so the date of measurement must be given. The term BP (before present) should never be used for luminescence ages, as BP has a specific meaning and is only relevant for radiocarbon ages.

In summary, two sets of measurements need to be combined to calculate a luminescence age (Fig 3), D_e , which is determined using luminescence, and the dose rate.

I.1 Abbreviations

a	annum (one year)
AAS	atomic absorption spectrophotometry, a technique used to analyse the chemical composition of materials
C	Carbon
D_e	equivalent dose – see Glossary
eV	electron-volt, a measure of energy: $1\text{ eV} = 1.602 \times 10^{-19}$ joule
Gy	Gray – see Glossary
ICP-MS	inductively-coupled plasma mass spectrometry, a technique used to analyse the chemical composition of materials
IRSL	infrared stimulated luminescence – see Glossary
K	Potassium
ka	kiloannum, a time period of 1,000 years
keV	kiloelectron volts: $1\text{ keV} = 1,000\text{ eV}$ (see eV)
LED	light emitting diode
Ma	megaannum, a time period of 1,000,000 years
MeV	megaelectron volt: $1\text{ MeV} = 1,000,000\text{ eV}$ (see eV)
NAA	neutron activation analysis, a technique used to analyse the chemical composition of materials
nm	nanometre ($1,000,000\text{ nm} = 1\text{ mm}$)

OSL	optically stimulated luminescence
P	palaeodose, another term used for equivalent dose (D_e)
ppm	parts per million ($10,000\text{ ppm} = 1\%$)
Ra	Radium
Rn	Radon
SAR	single aliquot regenerative dose, a sequence of luminescence measurements used to estimate the equivalent dose (D_e) of a sample
SI	Système International, an internationally recognised set of units used to define length, time, energy and other units of measure
Th	Thorium
TL	thermoluminescence – see Glossary
U	Uranium
XRF	X-ray fluorescence, a technique used to analyse the chemical composition of materials
$\mu\text{ m}$	micrometre ($1,000\mu\text{ m} = 1\text{ mm}$)

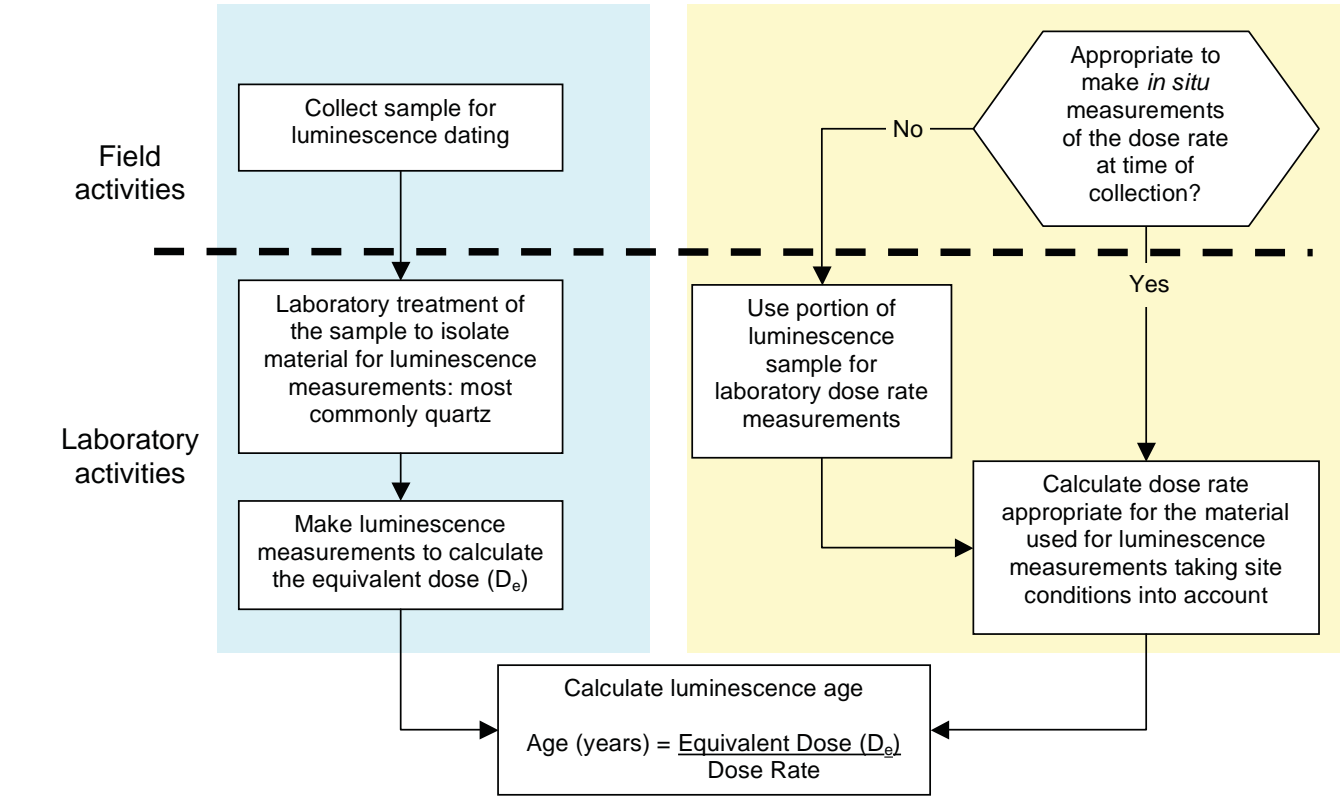


Fig 3 Flow chart showing how procedures to measure D_e (left) and the dose rate (right) are combined to calculate a luminescence age.

1.2 Common units for measuring length and time

Measurement of length is based on the SI unit of the metre.

	Abbreviation	length of one metre
metre	m	1m
centimetre	cm	100cm
millimetre	mm	1,000mm
micrometre	μ	1,000,000 μ m
nanometre	nm	1,000,000,000nm

The SI unit of time is the second(s), but this is not used in common parlance for long periods of time (for example an average year is 31,557,600,000 seconds). Thus for archaeological and geological purposes, the year is commonly used.

	Abbreviation	duration of one year
year	a	1a
thousand years	ka	0.001ka = 1a
million years	Ma	0.000 001Ma = 1a

2 The physical basis of luminescence

The analogy of a rechargeable battery shown in Fig 2 is a close one: D_e is a measure of the energy absorbed by the mineral grains and the dose rate is the rate at which the energy was delivered. In fact, at a sub-atomic level, the energy is stored by electrons becoming trapped at sites within the crystal structure – where they are normally forbidden to reside – but where they can be stored because of defects within the structure. An energy level

diagram illustrates the processes going on within the crystal (Fig 4). The interaction of radiation with the crystal provides energy to electrons that are raised to the **conduction band**. From here they can become trapped at defects (**trapping centres**) within the crystal. The electrons may be stored at these defects for some time. When an electron is released, it loses the energy that it gained during irradiation, and may emit part of that energy in the form of a single photon of light. In general, the deeper the defect below the conduction band, the longer the electrons remain trapped at that location without escaping at typical burial temperatures.

2.1 Thermoluminescence (TL)

The trapped electrons stored within minerals can be released in the laboratory using a number of methods that cause them to produce a luminescence signal. Heating the sample at a fixed rate from room temperature to between 450°C and 700°C releases the trapped electrons. The resulting signal is termed **thermoluminescence** (TL), and as shown in Fig 5 the signal is plotted as a function of the measurement temperature. Typically the TL signal (commonly referred to as a **glow curve**) comprises a series of peaks. Each peak may be due to a single type of **trap** within the mineral being measured, but more commonly the signal is a composite of several traps. Although it is not always possible uniquely to identify the source of the electrons, it is normally true that the TL signal observed at higher measurement temperatures originates from traps that are deeper below the conduction band. This is the case because more energy is required to

release electrons from deeper traps, and so this only occurs at higher measurement temperatures. A corollary of this is that electrons in deeper traps are more stable than those in shallower traps. For example, Fig 5 shows a TL measurement on an **aliquot** (a small sub-sample [c 1 to 5mg in mass] of quartz). The electrons in the trap giving rise to the TL peak observed at c 110°C are relatively unstable, and at room temperature have a mean lifetime of c 20 hours. This short lifetime makes them useless for dating. In contrast, measurements suggest that the electrons giving rise to the TL peak observed at c 325°C are stable over many millions of years. These two peaks could be thought of as derived from traps T_1 and T_2 , respectively, in Fig 4. The stability of the deeper trap has been confirmed by dating of sediments up to almost a million years old, and it is this trap that is the focus of most methods used for dating with quartz.

2.2 Optically stimulated luminescence (OSL)

A second means of releasing the electrons stored within minerals is by exposing them to light (Huntley *et al* 1985). This has become the most commonly used method. As soon as the stimulating light is switched on luminescence is emitted by the mineral grains. As measurement continues, the electrons in the traps are emptied and the signal decreases. The signal is termed **optically stimulated luminescence** (OSL) and Fig 6 shows data from an aliquot of quartz during optical stimulation. The signal initially decreases rapidly, and then at a slower rate. A similar signal is observed from feldspars, although a general observation is that the OSL signal from feldspars decreases more slowly than that from quartz. Unlike TL, the OSL data typically obtained (eg Fig 6) does not show which traps emitted the electrons. Thus, before making an OSL measurement, it is important to thermally pretreat the aliquot so that a signal is obtained from the group of traps of interest. This is achieved by heating the aliquot before measurement so that the shallow traps, whose electrons are unstable over the burial period (eg T_1 in Fig 4), are emptied, leaving only the electrons in deeper, stable traps (eg T_2 in Fig 4) – this heating is called a **preheat**, described in section 3.1.2.

The light used to stimulate the minerals is restricted to a narrow range of wavelengths so that this light can be prevented from reaching the sensitive light detector (the **photomultiplier tube**) used to measure the luminescence signal (Fig 7 page 8). This is because the light given off by the sample – the luminescence being measured – has to be observed at a different wavelength from the stimulation light. Blue light emitting diodes (LEDs) are commonly used for

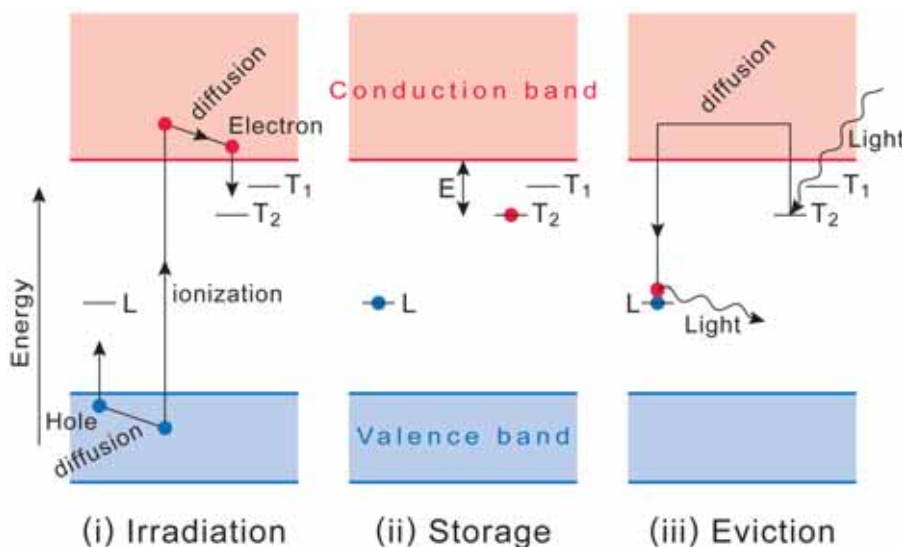


Fig 4 Energy level diagram illustrating the luminescence process: (i) radiation interacts with the crystal (ionization), pushing electrons into the conduction band and leaving 'holes' in the valence band; (ii) electrons become trapped at defect sites (T_1 , T_2 etc) and remain for a period of time (the deeper the trap below the conduction band (E) (eg T_2) the more stable the electron and the longer it stays trapped); (iii) crystal is stimulated by heat or exposure to light, releasing electrons, which recombine with holes at luminescence centres (L) and emit light photons = the luminescence signal (modified from Aitken 1990).

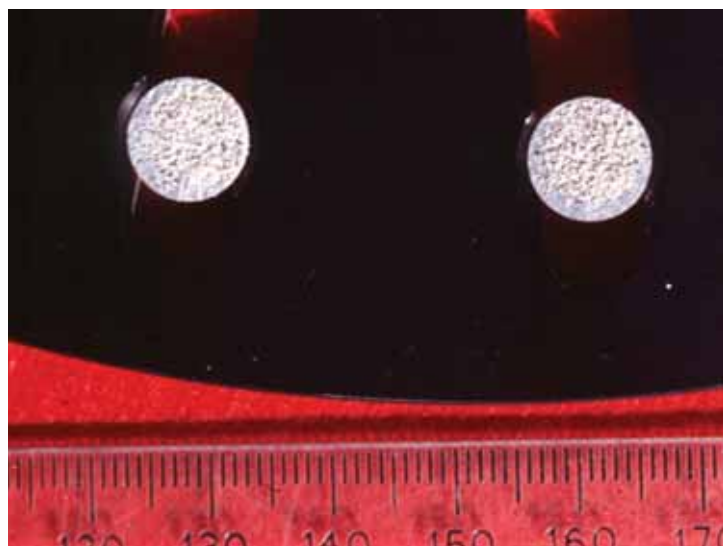
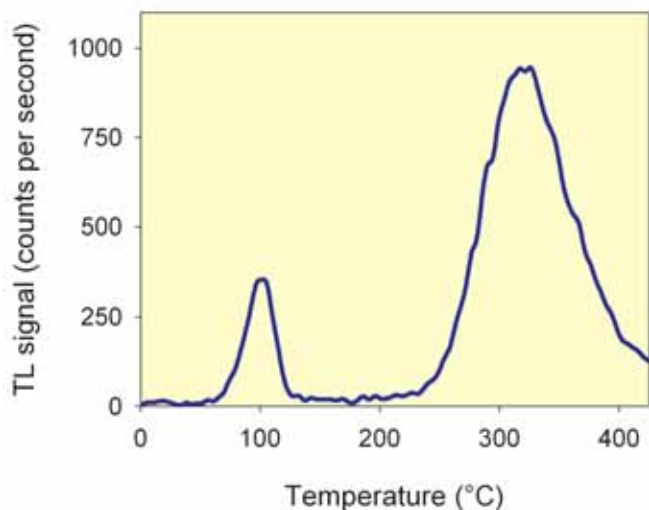


Fig 5 (left) Typical thermoluminescence (TL) signal from an aliquot of quartz as it is heated from room temperature to 450°C at a rate of 5°C per second. This sample had been exposed to a laboratory radiation source shortly before measurement, which induced the peak at about 110°C – this peak is only stable for a matter of hours at room temperature, so is not seen when natural samples are measured. The broad peak at c 325°C and the higher temperature shoulder are stable over many millions of years, and are used for dating. (right) Quartz grains (c 0.2mm diam) mounted on aluminium sample holders (9.8mm diam), ready for luminescence measurement (© Risø National Laboratory, Denmark).

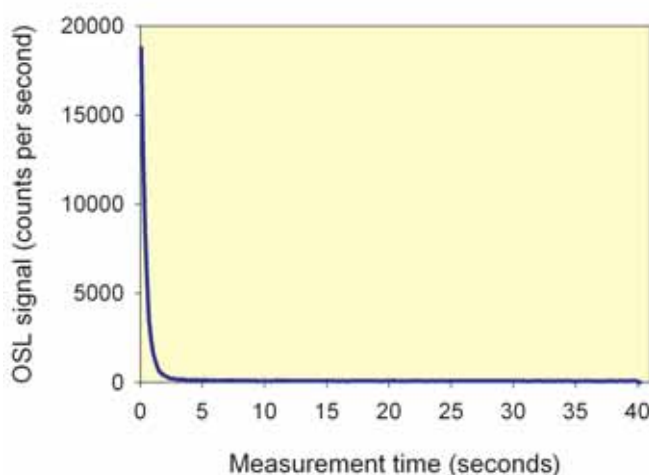


Fig 6 (left) Typical OSL signal observed from a quartz aliquot – the OSL signal drops rapidly as the trapped electrons in the quartz are used up to produce the luminescence signal. (right) An automated luminescence reader equipped with blue-light-emitting diodes (LEDs). When in use, the lid closes, leaving the sample holder in darkness. The blue LEDs are switched on to measure the optically stimulated luminescence (OSL) signal (© Risø National Laboratory, Denmark).

stimulation and will generate an OSL signal from both quartz and feldspar:

An alternative method of stimulation is to use LEDs that emit beyond the visible part of the spectrum in infrared. These are the type of LEDs used for remote controls in televisions, and for luminescence work those centred at 880nm have been used extensively. OSL signals are produced, but are more commonly referred to as **infrared stimulated luminescence (IRSL)**. IRSL is only observed from feldspars, with quartz not giving an IRSL signal when the sample is measured at room temperature. The fact that quartz does not emit an IRSL signal at room temperature can be exploited to provide a method for assessing the purity of quartz separated from feldspar for luminescence measurements.

2.3 Resetting of TL and OSL signals

The major advantage of OSL over TL is that the OSL signal is reset by exposure to sunlight much more rapidly than is the TL signal. This process is commonly known as **bleaching** and experiments in the laboratory show that after as little as 100s exposure of mineral grains to sunlight, the OSL signal from quartz is reduced to < 0.1% of its initial level (Fig 8 Page 8), but > 85% of the TL signal still remains. After several hours of exposure, > 30% of the TL signal remains but the OSL signal is almost one hundred thousands times lower. A similar situation exists for feldspars, with the TL signal being reduced by exposure to daylight much more slowly than the OSL (or IRSL) signal. The results shown in Fig 8 were obtained by placing a single layer of grains on a clean surface in the laboratory and exposing them to sunlight. In nature, this bleaching process is likely to be more complex, as mixtures of

mineral grains of different sizes and compositions are moved through the surface environment, and because the strength of daylight varies with the time of day and with the local conditions. In fact, as grains are moved around before their final deposition, they may be exposed to daylight more than once. In addition, grains may have a surface coating that reduces light penetration and thus causes slower bleaching. One of the major advances in luminescence dating in recent years has been the ability to make replicate measurements on sub-samples of the same material, and this provides a means for assessing whether the mineral grains were exposed to sufficient daylight for the OSL signal to be reset at deposition (see section 3.2).

2.4 Luminescence emission spectra

The luminescence emitted by minerals may occur at a variety of wavelengths. The lower

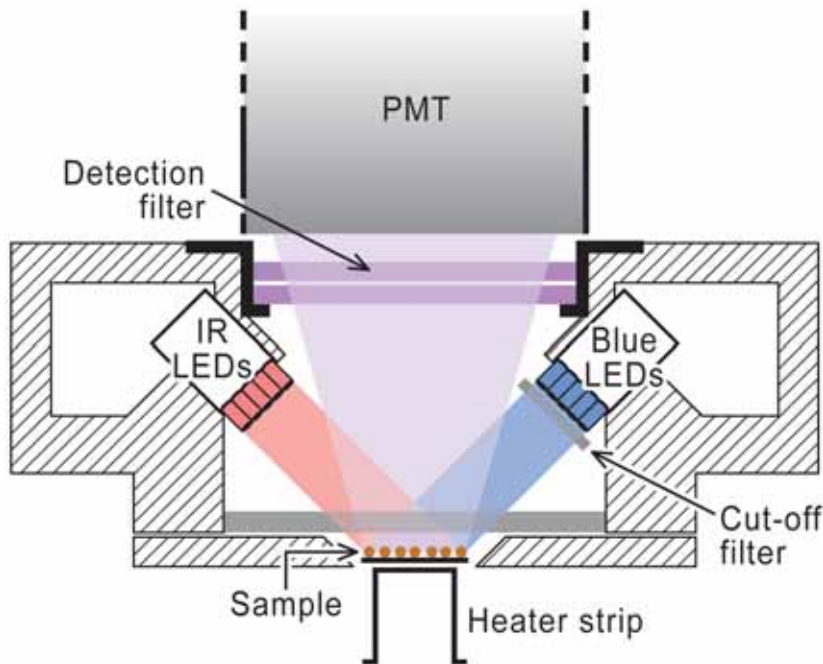


Fig 7 Luminescence emissions are detected by a highly sensitive light detector – a photomultiplier tube (PMT). The PMT lies above the sample, separated from it by glass detection filters. Stimulating the sample with heat from a metal heater generates a TL signal; light-emitting diodes (LEDs) give an OSL measurement – blue LEDs are commonly used for quartz, their emission range reduced by a cut-off filter (designed to block LED light from the PMT); infrared LEDs stimulate a signal from feldspar.

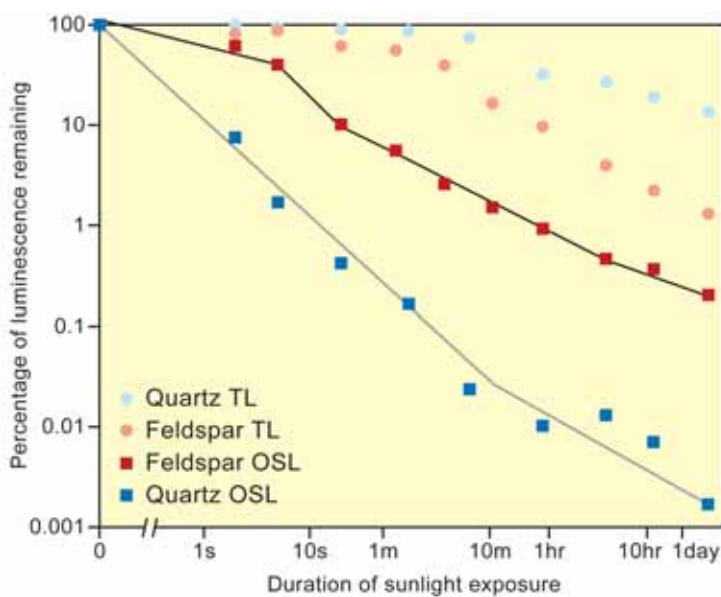


Fig 8 Reduction of TL and OSL signals from quartz and feldspar by exposure to light (redrawn after Godfrey-Smith et al 1988). Note that both axes have logarithmic scales.

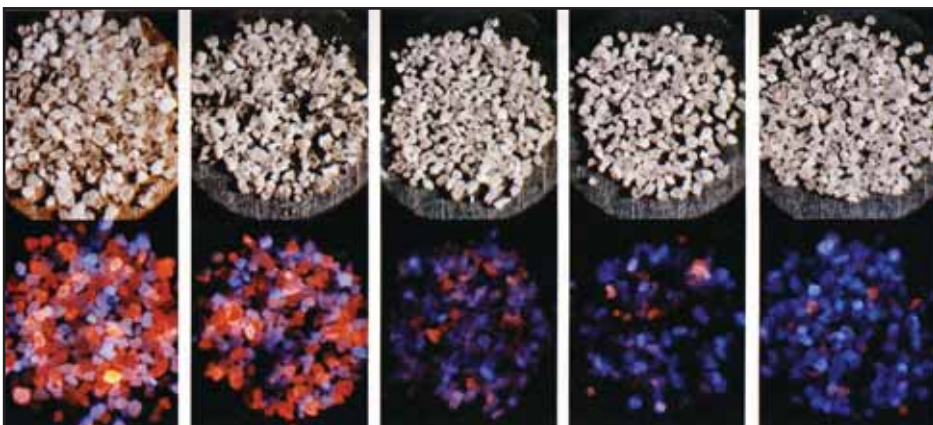


Fig 9 (upper row) Five quartz samples shown in room-light conditions (each metal disc = 9.8mm diam; quartz grains c 0.2mm diam). (lower row) Image, taken using highly sensitive photographic paper, of the thermoluminescence (TL) signal emitted by the grains being heated. Some grains emit most strongly in the orange–red part of the spectrum; others are strongest in the blue (from Hashimoto *et al* 1986).

panels in Fig 9 show photographs of the TL emitted by a number of aliquots of quartz, each comprising many hundreds of sand-sized grains (c 0.2mm diameter). One can see that some grains emit most strongly in the blue part of the spectrum while others emit in the red. More detailed spectral analysis of a range of quartz samples confirms that these two emissions (460–480nm and 610–630nm respectively), along with a series of emissions in the violet (360–420nm) are common to many types of quartz (Fig 10).

Feldspars cover a wide range of chemical compositions. Their luminescence emission spectra are more complex than those from quartz, with luminescence emitted at various wavelengths across the spectrum from ultraviolet to red and infrared (Fig 10).

Research and dating applications have focussed on looking at emissions from minerals in the blue and ultraviolet parts of the spectrum as most laboratories are equipped with instruments designed to look solely in these wavelength regions. Given the widespread use of blue LEDs emitting between 450nm and 490nm for optical stimulation, the most common filter used to reject this stimulation light is the Hoya U-340. This filter transmits emissions from c 280nm to 380nm (ie the near ultraviolet) (Fig 11), including the emission at 370nm (Fig 10), so that it can be detected by the photomultiplier tube.

2.5 Anomalous fading in feldspars

One of the main reasons for the focus in recent years on quartz for dating has been that, providing one works with electrons originating from a sufficiently deep trap, one can be confident of their stability. In contrast, analysis of feldspars has shown a more complex situation. As with quartz, electrons that are stored in traps that are deeper below the conduction band are calculated to be more stable than those from shallower traps. However, laboratory experiments have shown that electrons from deep traps in feldspars are less stable than expected – a situation called **anomalous fading**. This means that the size of the observed luminescence signal decreases as the sample is stored in the laboratory, and does so in an anomalous manner because the physical parameters of the trap suggest that it should be stable. There has been substantial debate in the discipline about the nature of anomalous fading, about whether it is a universal phenomenon shared by all feldspars, and whether it can be overcome. While this is an ongoing topic the current consensus is that many types of feldspar do exhibit anomalous fading, and also that the rate at which the signal fades varies from one sample to another.

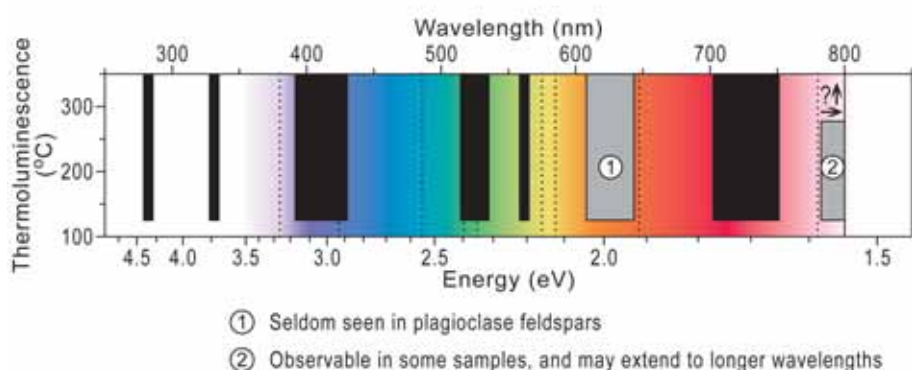
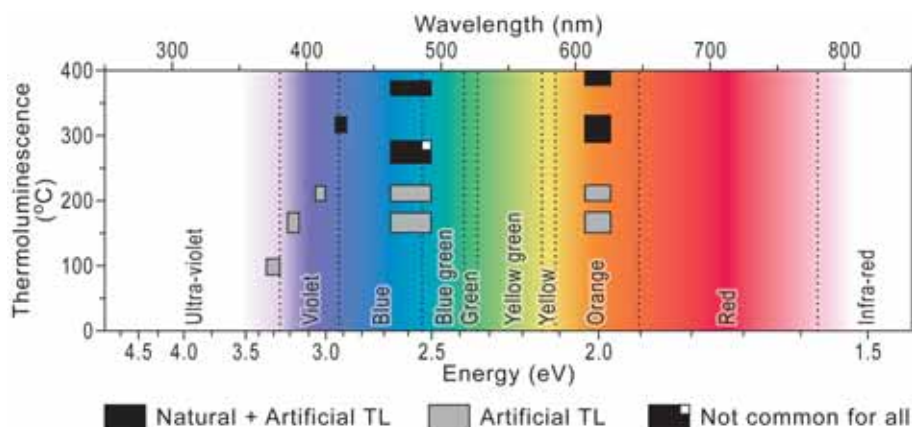


Fig 10 Quartz (above) and feldspars (below) emit luminescence at wavelengths ranging from ultraviolet through visible into infrared – dark bands show the strongest emissions. Quartz shows clear TL peaks – some are stable through geological time and found in natural samples; others are relatively short-lived (peaks > 200°C) and only found in artificially irradiated samples (modified from Stokes and Fattahi 2003).

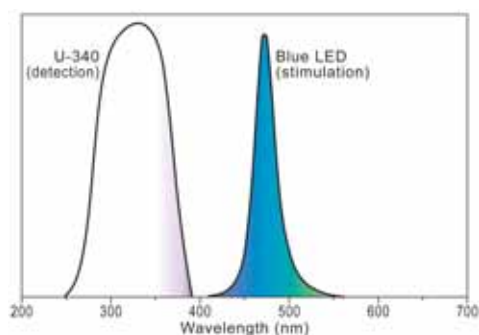


Fig 11 Blue LEDs, with emission centred at c 470nm, create a very bright source of light used to stimulate an OSL signal from quartz. The luminescence signal emitted from a sample is typically a million million (ie 1,000,000,000,000) times weaker than the LEDs. Such a weak signal is detected using a photomultiplier tube (PMT), filtering the PMT from the blue LEDs with a Hoya U-340 (which blocks light wavelengths > c 380nm).

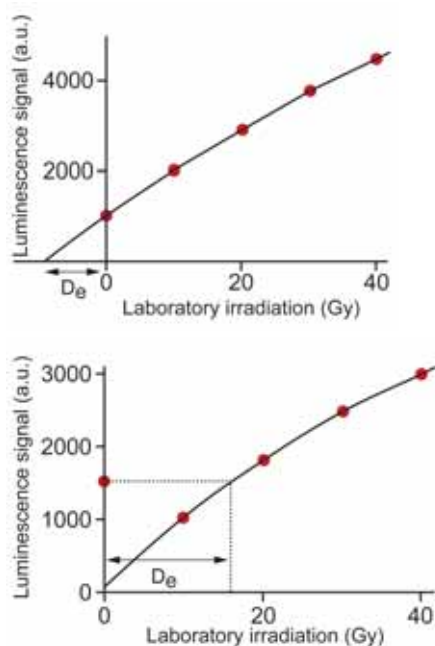


Fig 12 Two ways to calculate D_e from luminescence measurements: (above) 'additive dose' gives laboratory radiation doses on top of the signal acquired during burial, causing the luminescence signal to increase, to which a mathematical function can be applied – the function is extended until it intersects the x-axis, giving the estimate of the radiation dose the sample received during burial (D_e); (below) 'regeneration' measures the intensity of the luminescence signal from the natural radiation dose – shown as a horizontal dotted line. The sample's luminescence signal is reset by heating or exposure to light, and then given different laboratory radiation doses, characterising the growth of the luminescence signal with dose. D_e is calculated by finding the laboratory radiation dose that gives the same luminescence signal as that found in the sample when it was recovered.

Additionally, it is clear that luminescence signals emitted at different wavelengths may also fade at different rates.

Two broad approaches have been suggested to overcome the problem, in order to make it possible to use feldspars for routine dating. The first is to characterise the rate of anomalous fading, and then to apply some form of mathematical correction. The second is to look at different luminescence signals from feldspar and to try to find one that does not exhibit anomalous fading, eg red emission (Fig 10).

2.6 Other luminescence signals

Novel methods of obtaining a luminescence signal from minerals are constantly in development. Different procedures of luminescence production may be used, such as radioluminescence (RL), isothermal TL (ITL) and Thermally-Transferred OSL (TT-OSL). Some of these procedures have the potential to yield signals that may be useful in extending the age range of luminescence dating, but at the moment they remain experimental. Routine luminescence dating is based on the application of TL or OSL to heated materials, and OSL to sedimentary materials, and measurement is presently mostly made on quartz.

3 Measurement of D_e

In the application of luminescence to dating archaeological or geological materials, the objective of the luminescence measurements is to calculate the amount of radiation that the sample has been exposed to since the event being dated. It is the combination of this radiation dose and the dose rate (see section 4) that enables an age to be determined. Laboratory measurements are used to calculate the amount of laboratory radiation that is equivalent to that received during burial – the equivalent dose (D_e).

The amount of light emitted by a sample per unit radiation dose varies from grain to grain, depending on each grain's individual geological history. Thus one must use a set of laboratory measurements to calibrate the luminescence response of each sample, and use these to derive a measure of the dose received in nature (D_e). Two approaches have been widely used: additive dose and regeneration (Fig 12). Of these, the regeneration method is now the most frequently used.

In the regeneration approach, the luminescence signal originating from the unknown dose (D_e) is measured. All subsequent measurements are made after resetting the luminescence signal in the sample, by exposing a set of aliquots to sunlight or to

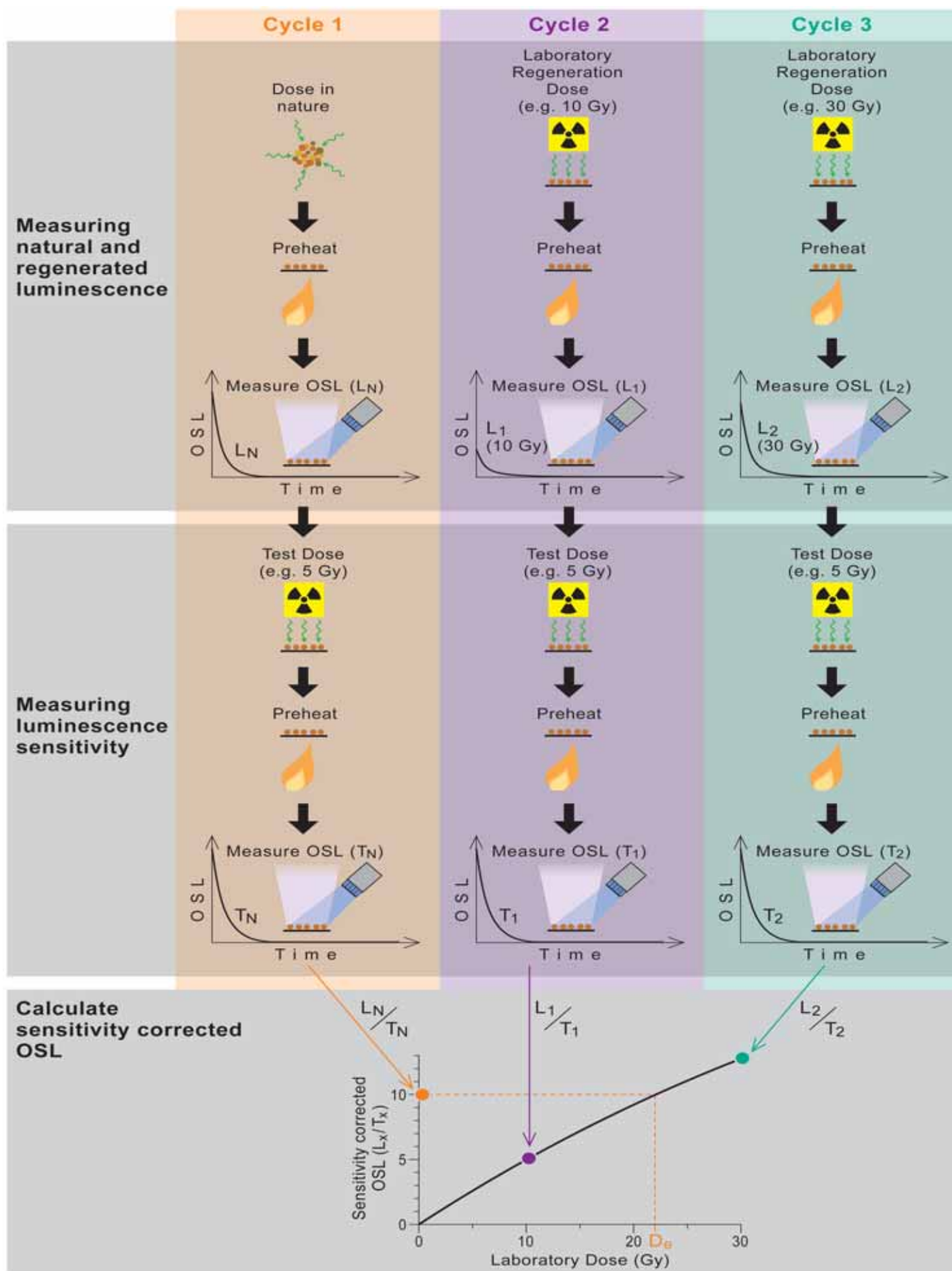


Fig 13 The single aliquot regenerative dose (SAR) procedure applied to quartz: the growth of the signal with dose is characterised by administering a number of laboratory doses (regeneration doses) of different sizes (10Gy, 30Gy, etc) and measuring the resulting OSL signals L_1 , L_2 , etc. After each measurement the luminescence sensitivity is measured by giving a fixed dose (here 5Gy) and measuring the resulting OSL signal (T_1 , T_2 , etc). The effect of changes in sensitivity can be corrected for by taking the ratio of the luminescence signal (L_x) to the response to the fixed dose (T_x). The plot of the sensitivity-corrected OSL (L_x/T_x) as a function of the laboratory dose (bottom) can be used to calculate D_e (here 22Gy) for that aliquot when the ratio of the initial measurements on the natural sample (L_N/T_N) is projected onto the dose response curve.

a controlled source of illumination in the laboratory (or, for samples heated in antiquity, by heating a set of aliquots). Known laboratory doses are then applied and this regenerated luminescence signal is measured (Fig 12). A curve is then produced characterising the growth of the luminescence signal from its residual level (after optical bleaching or heating). The aim of this approach is to determine the laboratory dose that generates a luminescence signal that matches the intensity of the signal originating from the natural sample.

The optimal method for determining D_e has been the subject of intense research activity in the last twenty years, and many different approaches are described in the academic literature. In 2000, the single aliquot regenerative dose (SAR) procedure was described (Murray and Wintle 2000). The method appears to be appropriate to many samples, has been shown to give accurate results when correctly applied, and has become the routine method of choice for measurement of D_e in quartz. A brief outline of some of the methods used to generate luminescence ages prior to 2000 is given in Duller (2004) and Lian and Roberts (2006).

3.1 SAR protocol

The procedure involved in single aliquot regenerative dose (SAR) measurements is illustrated in Figure 13.

It comprises a series of cycles. In the first cycle the OSL signal (denoted L) from the aliquot arises from the radiation dose to which the

sample was exposed in nature, and hence is given the term L_N . In the second cycle the aliquot is exposed to an artificial source of radioactivity in the laboratory. The OSL signal is then measured – this is the first laboratory regenerated signal, L_1 . Subsequent cycles measure L_2 , L_3 , etc as different regeneration doses (eg 10Gy, 20Gy, etc) are given to the aliquot. All of these measurements of luminescence are preceded by a preheat – heating the sample to a fixed temperature (normally between 160°C and 300°C) and holding it there for a short period of time (eg 10s). This procedure removes unstable electrons from shallow traps so that the OSL signal comes only from electrons that would have been stored safely through the burial period.

The brightness of these luminescence signals (L_1 , L_2 , L_3 etc) can be used to construct a **dose response curve** (Fig 12, lower). However, the luminescence sensitivity of the aliquot – the amount of light it emits for each unit of radiation to which it is exposed – changes depending on the laboratory procedures undertaken (eg the temperature and duration of the preheat treatment), and conditions during burial (eg ambient temperature). The second half of each cycle in Fig 13 addresses this problem by measuring the luminescence sensitivity. It is this second half of each cycle that was the major breakthrough in SAR: if such changes are not compensated for, then the calculation of D_e will be incorrect.

The luminescence sensitivity of the quartz is measured by giving a fixed radiation dose

(commonly known as a **test dose**) in the second half of each cycle and then by measuring the resulting OSL signal (T_N , T_1 , T_2 etc). The effect of any change in sensitivity can be corrected by plotting a graph, not of the luminescence signal (L_x) as a function of regeneration dose, but of the sensitivity corrected luminescence signal (L_x/T_x) (Fig 13). D_e can then be calculated, based upon this luminescence signal, corrected for any changes in sensitivity that may have occurred.

The SAR protocol has been widely applied to both heated quartz and unheated quartz (Wintle and Murray 2006) and been shown to give ages consistent with independent age control (Murray and Olley 2002). Fig 13 illustrates the idea behind the SAR procedure and shows three cycles of measurement. In practice, more cycles would be used, typically between five and ten in total (Fig 14). These additional cycles are used to define the dose response curve using a variety of regeneration doses, and to enable a number of checks (described below) to be made on the behaviour of each aliquot (Wintle and Murray 2006). It is crucial that these checks are made, as it is known that although SAR does work on most quartz samples, it does not work on all. In particular, some dim samples may not be appropriate for analysis using SAR because they lack the part of the OSL signal that gives the rapid decrease in signal observed during the initial part of the OSL measurement (Fig 6).

3.1.1 Recycling test

Once the dose response curve is defined as described above, it is routine to repeat the

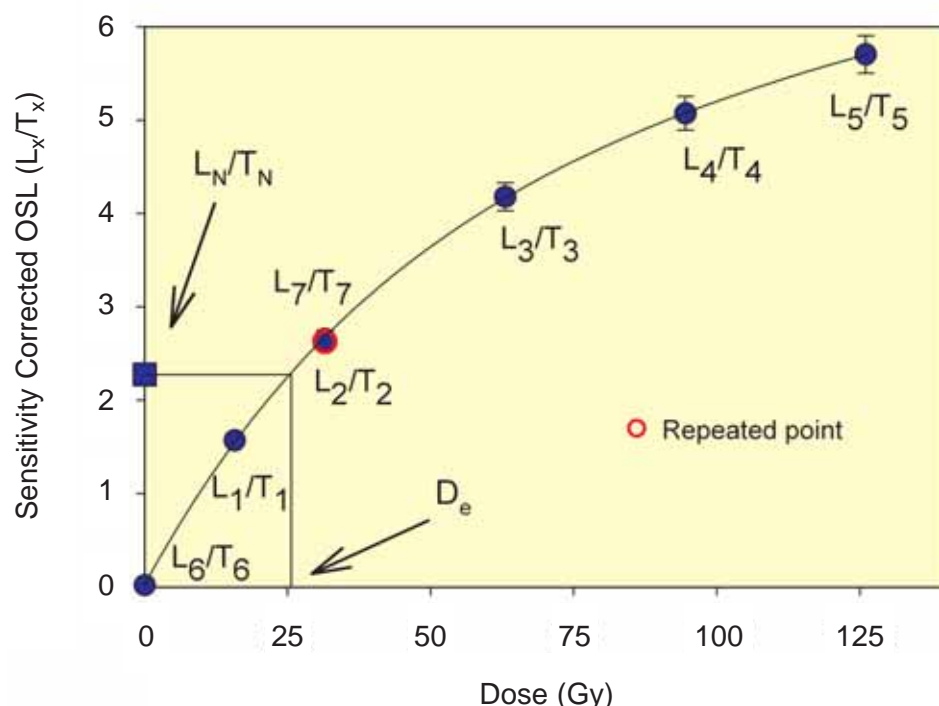


Fig 14 SAR dose response curve for a quartz aliquot from Kalambo Falls, Zambia, measuring eight cycles: first cycle – the natural signal (L_N) and its response to a test dose (T_N), enabling calculation of the sensitivity-corrected OSL (L_N/T_N); next, regeneration doses of 16, 32, 64, 96, 128 and 0Gy give sensitivity-corrected values L_1/T_1 , L_2/T_2 , L_3/T_3 , L_4/T_4 , L_5/T_5 and L_6/T_6 (these data define the dose response curve for this aliquot). By interpolating the value of L_N/T_N onto the dose response curve, D_e can be calculated; in this case 25.6 ± 1.3 Gy). The final cycle repeats the measurement for a regeneration dose of 32Gy (L_7/T_7). The ratio of the two measurements for 32Gy (L_7/T_7 divided by L_2/T_2) is known as the recycling ratio, and for this aliquot has a value of 0.99 ± 0.05 – close to the ideal value of 1.00, and well within the accepted range of 0.9 to 1.1.

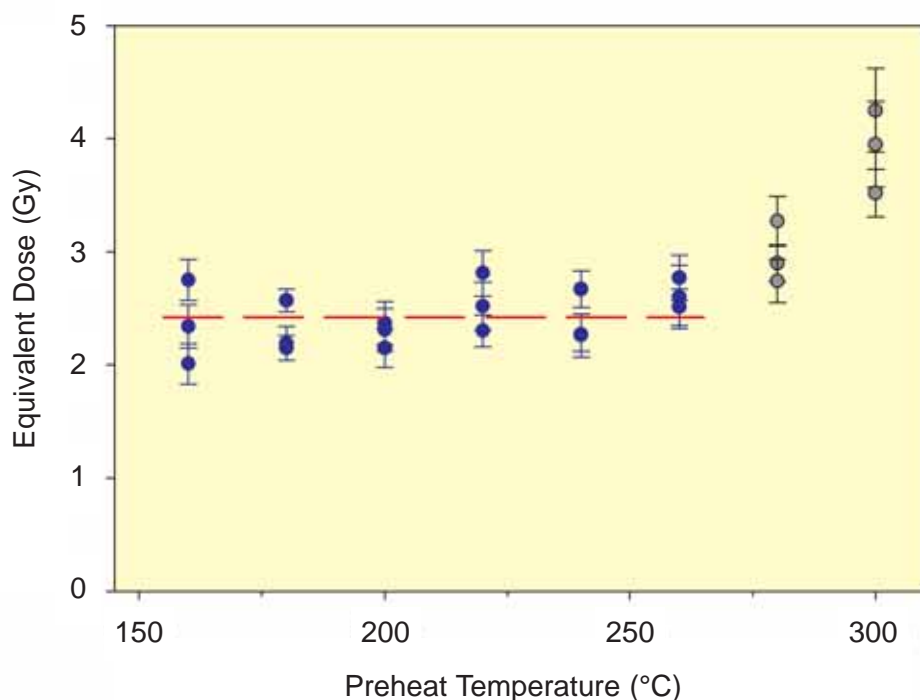


Fig 15 Preheat plot, showing D_e (and associated measurement errors) for beach sand buried beneath gravel at Dungeness Foreland, measured at preheat temperatures from 160°C to 300°C, for 24 aliquots (Roberts and Plater 2005; 2007). A plateau in D_e values extends from 160°C to 260°C (blue symbols) and the average value (dashed red line) was used to calculate the age of the sample ($3850 \pm 180a$; Aber73/BH-3/2). At higher preheat temperatures D_e increases (grey symbols), probably due to thermal transfer; so these data are excluded.

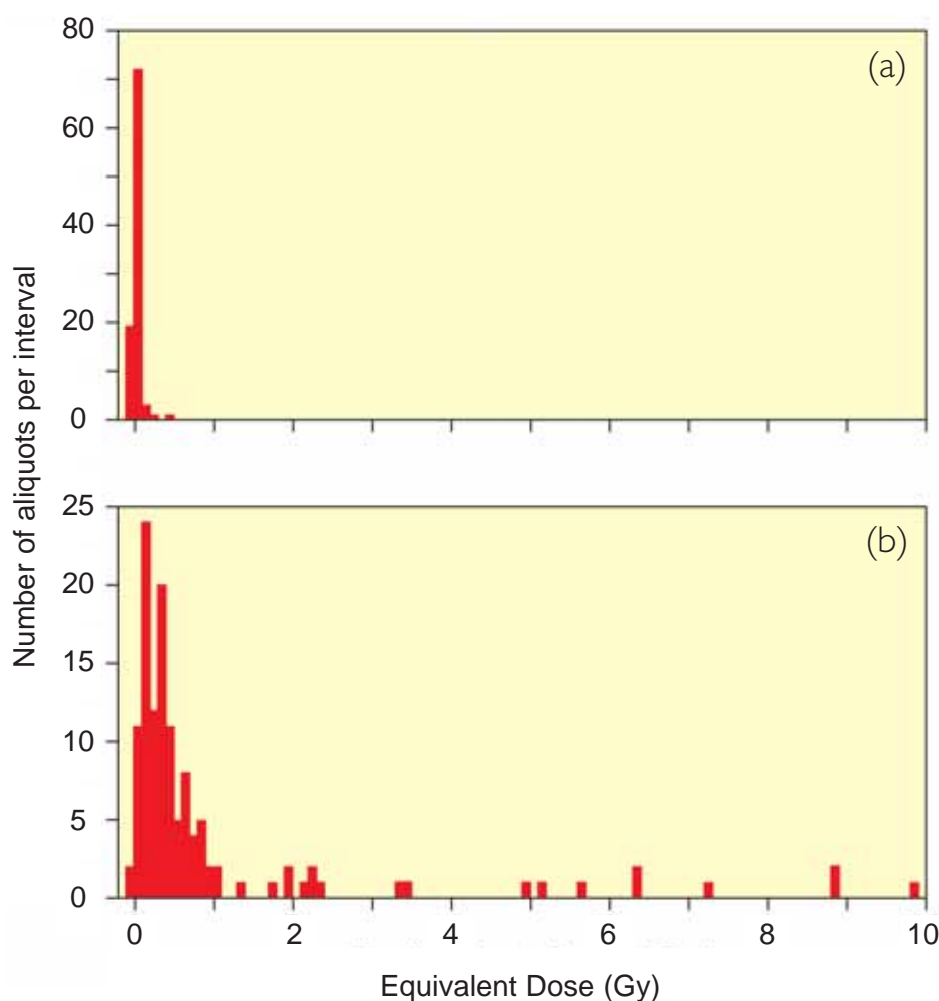


Fig 16 Replicate D_e measurements for two contrasting samples: (a) 31 measurements of a beach sand sample (Aber73/BH1-1) from Dungeness, all giving similar values. Since the samples belong to a single population, the mean D_e can be used for age calculation ($3.70 \pm 0.06Gy$); (b) 44 measurements for a sample of fluvial sand from Crete (Aber60/AN2002/4), showing large D_e variability. Using the average value of D_e to calculate a luminescence age would give a meaningless result. The data for each sample are shown as a histogram, and each D_e value (ranked in increasing order) is a point with an error bar of analytical uncertainty.

measurement of the luminescence signal resulting from one of the radiation doses. If the procedure is working appropriately, and the SAR corrects for changes in the sensitivity of the sample, then the luminescence signals (L/T) of the repeated dose (eg 32Gy in Fig 14) should be the same. The ratio between the two sensitivity-corrected luminescence signals is the **recycling ratio** – ideally 1; in practice, a ratio between 0.9 and 1.1 is deemed acceptable. Values greater than or less than these limits suggest that the procedure or the sample are inappropriate, and therefore that the results from that aliquot should be rejected.

3.1.2 Preheat test

Before each luminescence measurement, the aliquot is heated between 160°C and 300°C for 10s to remove unstable electrons. The appropriate preheat temperature is determined by making measurements of D_e using a range of temperatures. Fig 15 shows the results for one sample, measuring D_e for 24 aliquots: three aliquots with a preheat of 160°C, three at 180°C, three at 200°C etc to a maximum of 300°C. This procedure shows that the same D_e value is obtained for temperatures from 160°C to 260°C. At temperatures > 260°C D_e increases, probably because of **thermal transfer**.

A strength of the SAR procedure is that it works with a range of temperatures. Nevertheless, it is important to test one or two samples from within a suite of samples to determine if a suitable preheat is achievable.

3.1.3 Dose recovery test

One of the most powerful tests available – commonly undertaken with SAR – is the dose recovery test. This involves removing the trapped electron population from the sample, thus mimicking the resetting of the sample at the time of the event being dated.

For sediments this involves exposing an aliquot of naturally irradiated grains to daylight or to an artificial light source, irradiating the aliquot with a known dose (eg 10Gy), and then treating this dose as an unknown. The SAR procedure is applied. If it is appropriate then the value of the calculated dose should match the known laboratory dose. It is common to choose a known laboratory dose close to the value of D_e measured for the sample. In this way, complications introduced as samples become close to saturation at high dose values – or difficulties arising as one approaches the detection limit of the instrument for low dose values – are assessed implicitly. If a sample fails a dose recovery test then it is unlikely that the D_e calculated for the sample will be correct.

3.2 Replicate measurements of D_e

Single aliquot measurements reduce the time involved in determining D_e by automating much of the procedure; and they make it feasible to make replicate measurements of D_e for each sample. Thus it is routinely possible to assess the reproducibility of D_e within each sample. Replicate determinations of D_e would be expected to form a normal or log-normal distribution where three conditions hold true: (1) the sample contains grains that were completely bleached at deposition; (2) variations in the annual dose due to small-scale differences in the concentrations of uranium, thorium and potassium are small; and (3) no post-depositional mixing has occurred. In this situation, averaging the replicate D_e measurements gives an increase in the precision of the age calculated (Fig 16a).

If replicate measurements of D_e are not similar (as in Fig 16b) then this implies that the sample is more complex, and requires further investigation. The most common cause of such large scatter is that the trapped electron population in the different mineral grains of the sample was not completely reset during the event being dated – this is known as **incomplete bleaching**. The D_e values obtained for quartz from a modern sand dune form a tight cluster close to 0Gy (Fig 17a). However, when rivers transport sediments not all the mineral grains are exposed to sufficient daylight before deposition to reset their signal (Fig 17b). Thus the D_e that is measured in the laboratory will be the sum of the dose acquired since the sample was deposited, plus the residual signal remaining at the time of deposition. In such situations, the best estimate of the dose that has been acquired since the event of interest will be given by those aliquots with the lowest D_e values. Statistical models have been developed (Galbraith *et al* 1999; Galbraith 2005), to deal with such situations, and can be applied where the cause of the distribution in D_e values can be understood. While much can be learned from analysis of the distribution of D_e values alone, it is always important to consider the depositional context from which a sample has been collected when assessing the most likely cause of scatter in the distribution of D_e values.

The second potential cause of D_e variation is dose rate differences in different parts of the sample – different parts of the sample have received different radiation doses during the period of burial. Fortunately, such variation in microdosimetry can normally be avoided by careful sample selection. Situations where this problem is likely to be most severe are those where the sediment contains a small proportion of highly radioactive grains – for

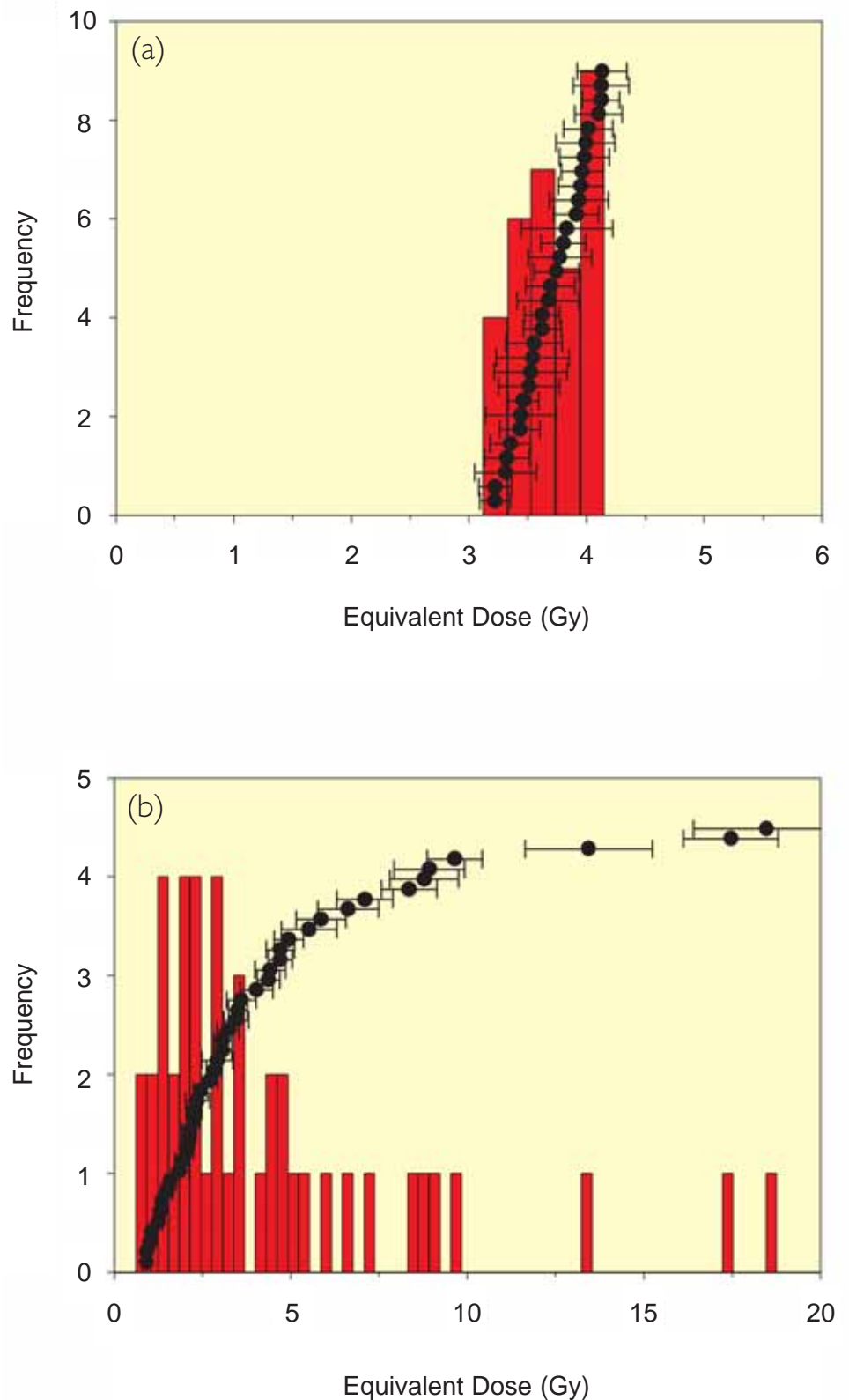


Fig 17 Histograms of D_e values for two samples, demonstrating the potential problem of dating fluvial samples. Aliquots containing between 60 and 100 quartz grains (125–180 μ m diam) were analysed: both samples should give D_e values close to zero, as they were collected from sites of current deposition. (a) Sand dune – D_e values close to zero indicate that it was exposed to sufficient daylight at deposition to reduce the luminescence signal to near zero. In contrast, (b), from a fluvial environment, produced many D_e values significantly greater than zero, implying that only some of the grains in this sample were exposed to sufficient daylight (modified from Olley *et al* 1998).

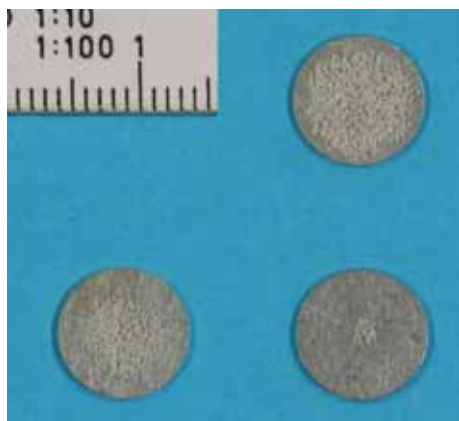


Fig 18 Aliquots for luminescence measurements may contain different numbers of grains: each metal sample disc is 9.8mm diam; changing the proportion of the disc surface used varies the number of quartz grains (c 0.2mm diam) (upper right c 1000 grains, bottom left c 200, bottom right c 20) (photograph by H Rodnight).

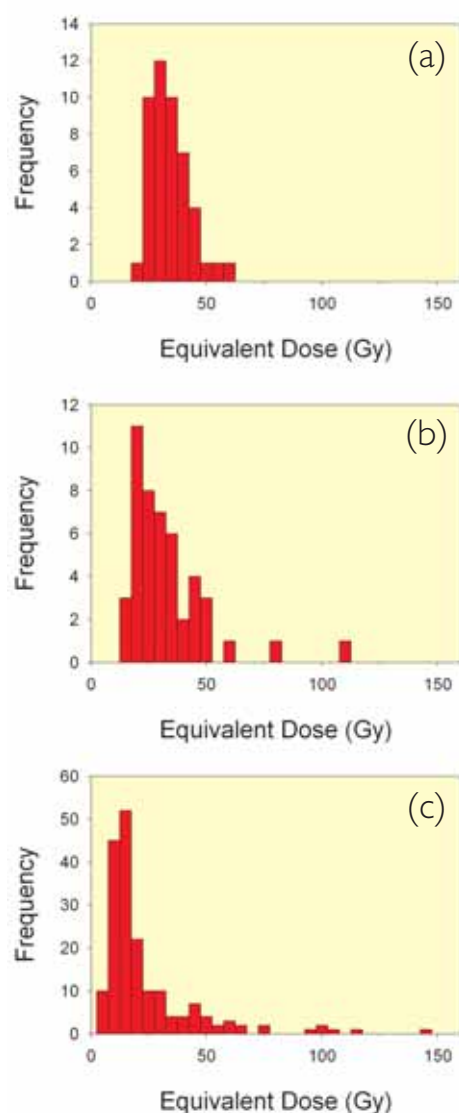


Fig 19 Potential effect of changing the number of grains in an aliquot – three histograms of D_e results for one sample using different numbers of grains in each aliquot: (a) c 1000 grains, (b) c 200 grains and (c) single grains. Many grains in aliquot (a) averaged out differences, masking the real variation; as the number of grains is decreased (b and c) the variability in the D_e becomes obvious. This sample was not completely bleached at deposition, so an age based on the large aliquots shown in (a) would have been an overestimate.

instance zircons – or where a sample is very heterogeneous, eg owing to the presence of gypsum with low radioactivity, or wood ash with relatively high radioactivity from a high potassium content.

Another situation that might produce a wide range of D_e values is a sediment sample whose grains are of different, mixed ages. Mixing might have occurred in antiquity (David *et al* 2007), possibly by human activity, or it may have occurred during sampling. Where samples of different ages are closely juxtaposed, for example, more than a single context might have been sampled in error (Jacobs *et al* 2008).

3.3 Aliquot size

In situations where different mineral grains have different D_e values, the results of single aliquot analyses will depend on how many grains are contained in each aliquot. In typical luminescence measurements, sand-sized mineral grains c 0.2mm in diameter are measured. For such a grain size it is possible to make single aliquots containing about a thousand grains, hundreds of grains, tens of grains (Fig 18) or even a single grain.

Fig 19a shows the result of making replicate D_e measurements on a sample. D_e values obtained are relatively similar. These measurements were made using aliquots of c 1,000 grains. Measurements of the same sample made using aliquots with c 200 grains show more variation in D_e (Fig 19b), and measurement of single grains increases the variation further (Fig 19c). This sample consists of grains with different D_e values, as is clearly shown in the single-grain results. The measurements on aliquots with hundreds or thousands of grains are masking these variations by averaging the results from many grains. Thus, if a sample is thought to be affected by incomplete bleaching or mixing, then making replicate measurements on aliquots with relatively few grains, or even with single grains, should be used to test this possibility. The spread in D_e shown in Fig 19a, as obtained in routine measurements using aliquots of c 1,000 grains, would not necessarily cause suspicion of incomplete bleaching.

3.3.1 Single-grain OSL measurements

In the last five years, measurements on single, sand-sized grains have become feasible using a focussed laser. This laser replaces the blue LEDs used for optical stimulation in conventional measurements, and can be directed so that it stimulates each individual grain in turn, making OSL signal measurement possible. On each 9.8mm diameter aluminium

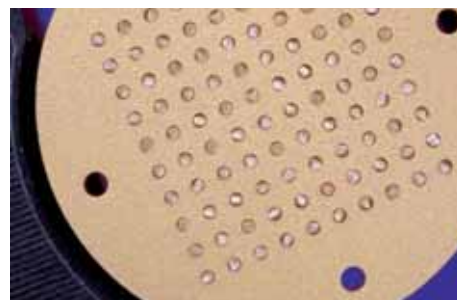
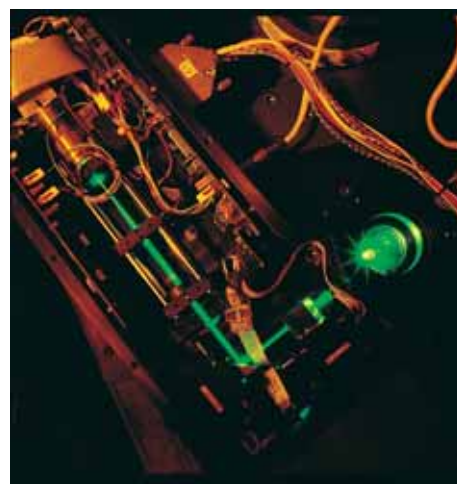


Fig 20 (top) Luminescence measurements made by focussing a green laser beam on the sample (beam diam c 0.02mm when it strikes a grain). (bottom) Individual quartz grains (0.2mm diam) held in an array of 10 x 10 holes drilled in the surface of the 9.8mm diam sample holder. The laser beam is moved using mirrors to strike one grain at a time, giving an OSL signal (© Risø National Laboratory, Denmark).

disc, 100 individual grains can be held, each in its own hole, 0.3mm in depth and 0.3mm in diameter; drilled into the surface of the disc (Fig 20).

These measurements have demonstrated that for most samples of quartz, the luminescence signal that is observed is dominated by the signal from a relatively small proportion of the grains. Typically, 95% of the light that is observed as OSL originates from less than 5% of the grains. The reason for this variability in the brightness of individual mineral grains, even though they are collected from the same sample, is not well understood, but is thought to relate to the original conditions of formation of the quartz grain. However, this observation has two important implications. First, that when single-grain measurements are made, many of the grains do not emit sufficient OSL to enable D_e to be determined. It is not uncommon for 5% or less of the grains that are analysed to yield useful data. Second, this finding implies that even though aliquots may physically contain several hundred grains, the OSL signal observed is likely to originate from a much smaller number of grains. Thus measurements using small aliquots, typically consisting of many tens of grains each, may

yield results that are similar to those from single grains. Such measurements may thus reduce the need for time consuming single-grain analyses and the requirement for a laser stimulation system.

3.3.2 Displaying D_e datasets for samples

The large variation in the brightness of individual grains also has an impact on the precision with which D_e can be determined. Where a grain does not emit a bright luminescence signal, then the precision of the measurements may be limited, while brighter grains give more precise D_e estimates.

For example, imagine a sample that has been given a radiation dose of 10Gy. A bright grain from this sample may yield a D_e of 9 ± 1 Gy while a dim grain may give 15 ± 5 Gy. Both values are consistent with the dose of 10Gy given to the sample. We could also imagine another sample that consists of a mixture of grains with different doses. Two grains from this sample could give D_e values of 9 ± 1 Gy and 15 ± 2 Gy. In this latter case, the two grains would give ages that are inconsistent with each other. If we plotted these two sets of results on a histogram, such as those shown in Fig 19, they would appear to be identical, even though statistically they are different – one is consistent with both grains having received the same radiation dose, while the other is not. This is because in a histogram there is no way of taking into account the uncertainty on the values being shown.

An alternative method of displaying this type of data is a radial plot (Galbraith *et al* 1999; Galbraith 2005). This method is widely used for displaying single-grain D_e data. Figure 21 shows such a radial plot. On the diagram, each point represents the D_e from a single grain of quartz, in this case 289 grains. The horizontal axis shows how precisely the D_e value is known, with more precise values on the right-hand side of the graph. The precision can also be expressed as a relative error. Thus a value of 10 ± 2 Gy has a relative error of $2/10$ or 20%.

The vertical axis, shown on the left-hand side of the diagram, is called the standardised estimate. This shows how different the D_e value for each grain is from some reference value. The calculation of this is complex (details are given in Galbraith 2005), but the important property of the diagram is that if one draws a straight line from the origin (the point where the standardised estimate and the precision are both zero) towards the curved axis shown on the right-hand side, any point on that line represents a grain with the same D_e value. The precision of D_e will vary, but D_e itself will be

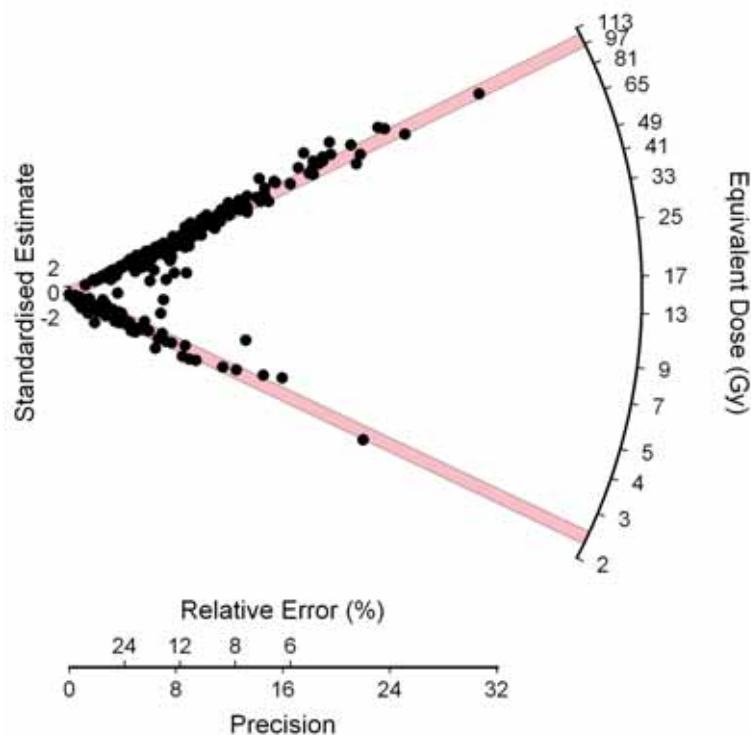


Fig 21 Radial plot of D_e values for 289 single quartz grains from Sibudu Cave, South Africa. The data form two groups: one with an average value of 101Gy (upper band), and one of 2.4Gy (lower band). The upper band contains 71% of the grains and gives a luminescence age of 35 ± 2 ka, as expected for this sample from the Middle Stone Age (MSA). The 29% of the grains in the lower band give an age of 0.93 ± 0.04 ka, consistent with the South African Iron Age (IA). At Sibudu Cave the IA immediately overlies the MSA, and the single-grain analysis shows that in sampling the two contexts were inadvertently mixed (Jacobs *et al* 2008).

the same. Thus the curved, radial axis on the right-hand side can be labelled so that one can read off the D_e values. By convention, this axis is logarithmic, as in Figure 21.

The data shown in Figure 21 clearly fall into two distinct groups. The upper group forms a band that points towards the radial axis and intersects with it at a value of 101Gy. The site in South Africa from which this sample was collected contains sediments from the Middle Stone Age, and these grains yield an age of 35 ± 2 ka. However, collection of this luminescence sample inadvertently cross-cut into a different sedimentary unit, dating to the Iron Age. The second band of grains, intersecting the right-hand axis at a value of 2.4Gy, is the result of this mixing, and would give an age of $c 0.93 \pm 0.04$ ka (Jacobs *et al* 2008).

Single-grain measurements of D_e are very powerful, and can help to elucidate the causes of complex D_e distributions (Jacobs and Roberts 2007). However, such measurements are extremely time-consuming, and are frequently made difficult by the low luminescence signal levels encountered. Thus at present they are far from routine, and results obtained by using aliquots consisting of tens or hundreds of grains may be equally appropriate. In the future it is likely that single-grain methods will become more commonplace.

3.4 TL and the plateau test

The SAR method described in section 3.1 is used increasingly for measuring D_e for sediments using the OSL signal from quartz. OSL is appropriate for dating sediments as it is more rapidly reset by exposure to daylight than TL is (Fig 8). For materials that were heated in antiquity – such as pottery, burnt flints and brick – one can use either OSL or TL signals.

One advantage of TL measurements is that additional information is contained in the TL glow curve (Fig 5). In a TL measurement, luminescence signals emitted at higher temperatures tend to originate from traps that are deeper below the conduction band (Fig 4), and hence are expected to have been stable over longer periods of time. A useful application of this is the plateau test: D_e for the sample can be calculated using the intensity of the TL signals measured at different temperatures (Fig 22 page 16). At low temperatures, the ratio of the natural signal divided by the laboratory-irradiated signal is very small, as the traps are unstable over geological time. As the temperature increases, so too does this ratio, until above some temperature, typically $c 300^\circ\text{C}$, a plateau is observed. The observation of a plateau confirms that above this temperature range the traps are sufficiently stable to accurately record the radiation dose acquired over time.

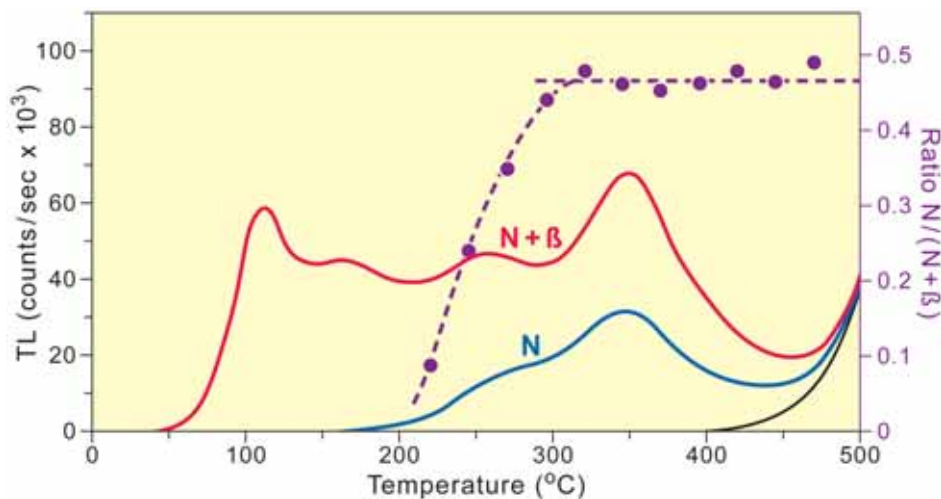


Fig 22 A plateau test, showing the natural TL signal (N) from one aliquot and the TL signal from a different aliquot after it had been irradiated with a beta dose in the laboratory to increase the signal (N+β) – the dots joined by the dashed line show the ratio of the two TL signals. The ratio is low at low temperatures and increases with higher temperatures; above 300°C the ratio becomes stable at c 0.47 (modified from Aitken 1985).

Additionally, when dating burnt flints, obtaining a plateau demonstrates that the material was heated sufficiently in antiquity to reset the sample. Data from the temperatures over which the plateau is observed are used to obtain D_e , which then forms the basis for calculating the time since heating in the past.

4 Measurement of dose rate

The application of luminescence to dating archaeological or geological materials relies on determining two quantities. The first is the amount of radiation absorbed by the sample during the period since the event being dated, measured as D_e . To determine the age of the sample in years, D_e has to be divided by the radiation dose received by the sample each year – the dose rate.

There are four types of environmental radiation: **alpha particles (α)**, **beta particles (β)**, **gamma rays (γ)** and **cosmic rays**. The first three originate from naturally occurring elements in the sample itself and its surroundings. The most important of these sources are radioactive isotopes of uranium (U), thorium (Th) and potassium (K). The radioactive isotope of potassium is ^{40}K , which decays to ^{40}Ca (calcium) or ^{40}Ar (argon) by the emission of beta particles and gamma rays. Both ^{40}Ca and ^{40}Ar are stable.

Uranium and thorium are more complex: ^{238}U decays to form ^{234}Th , which is itself radioactive, and will decay to form protactinium (^{234}Pa), and so on until a stable isotope of lead (^{206}Pb) is reached (Fig 23). Such a chain of isotopes is called a decay series. Similar decay series exist for ^{235}U and for ^{232}Th . The U and Th chains emit alpha, beta and gamma radiation and contribute to the total radiation absorbed by samples (Fig 24).

Cosmic rays originate from sources far out in the universe. They are a type of electromagnetic radiation similar to gamma rays derived from U, Th and K, but typically with much higher energy. The Earth is shielded from most cosmic rays by the atmosphere and by the Earth's magnetic field. However, a proportion does reach the Earth's surface and penetrate sediments. The radiation dose received decreases with depth, particularly rapidly in the uppermost metre.

Alpha, beta and gamma radiation is also absorbed and attenuated by the surroundings. Each type travels different distances in materials. Alpha particles are, atomically speaking, large objects, and typically only travel a few hundredths of a millimetre through sediments; beta particles are electrons, and will travel a few millimetres or more, while gamma rays may penetrate as much as 0.3m (see inset to Fig 24).

Two approaches can be used to measure the annual radiation dose provided to a sample from the radioactive elements surrounding it: measuring the concentration of U, Th and K, and using these concentrations to calculate the radiation dose received by the sample; or directly counting the emission of radiation. The better approach will depend upon the type of sample being dated and on practical considerations (primarily those of access). The different distances travelled by alpha, beta and gamma radiation directly influence the volume around the sample that needs to be measured.

4.1 Chemical methods

A range of geochemical methods exists to measure the concentration of the key elements responsible for delivering the

radiation dose – U, Th and K. For sediments and bricks, the dose rate from the surroundings can be measured from a finely ground sub-sample of the sediment itself. For pottery and burnt flints, both the sample and the sediment surrounding the sample need to be analysed.

Of the three elements measured, K occurs in the greatest concentration, typically 0.5% to 3% by weight. It can be measured using a range of methods, including atomic absorption spectrophotometry (AAS), flame photometry, X-ray fluorescence and inductively coupled plasma mass spectrometry (ICP-MS). In all cases a precision of c 5% or better would be expected.

U and Th occur in much lower concentrations, typically in the range of 1ppm to 10ppm. The range of measuring methods is more limited, but includes ICP-MS and neutron activation analysis (NAA).

Three major issues may arise with these measurements: (1) Concentrations of these elements may be close to the detection limits for some analytical facilities; minimum detectable limits are often quoted, and one would want these to be several times smaller than the concentrations being measured. (2) Sample preparation – ICP-MS and many other methods rely on dissolving the sample in strong acids to produce a solution that can be analysed; in some samples, U and Th may be associated with highly resistant minerals – if these are not completely dissolved during the analysis then the measured values will be underestimated. (3) These measurements are made on sub-samples of only a few mg, and such a small sample may not be representative of the sediment or brick being used for dating.

Once the concentrations of these three elements are known, conversion factors enable the calculation of the radiation dose rate (Adamiec and Aitken 1998). For example, 1% potassium in sediment will produce a gamma radiation dose rate of 0.243Gy per thousand years (Gy/ka), a beta dose rate of 0.782Gy/ka, but no alpha dose rate, as the decay of ^{40}K does not result in the emission of alpha particles. Adding together the alpha, beta and gamma dose rates gives the total radiation dose rate.

This approach makes a number of assumptions. For U and Th decay chains the concentration measured is that of the parent isotopes. Figure 23 shows the decay of ^{238}U . Radiation is emitted at all stages of the chain, but the chemical methods described above will only measure the concentration of U – they do not

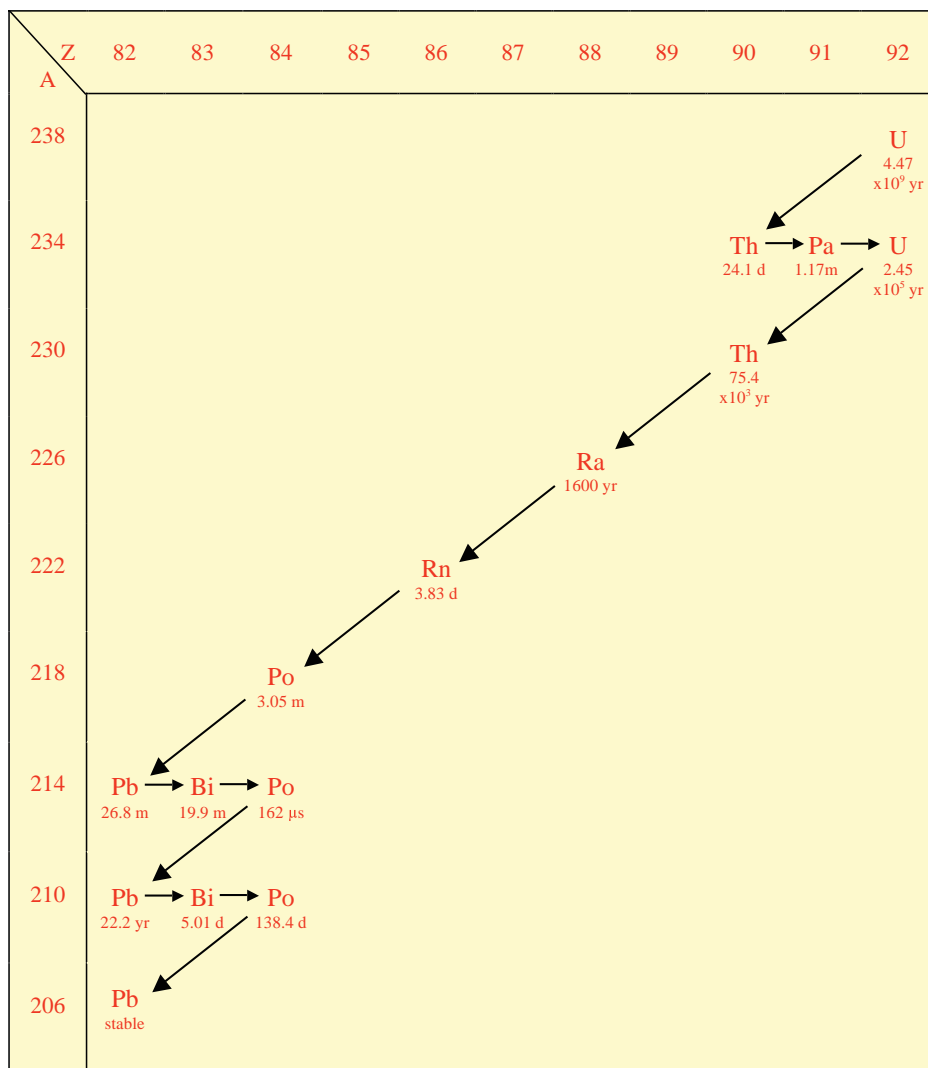


Fig 23 The decay chain of ^{238}U ; arrows show the sequence of isotopes produced by emission of a beta particle (horizontal arrows) or an alpha particle (diagonal), with half-lives varying from hundreds of thousands of years to less than a second (Z = atomic number; the number of protons in the nucleus and hence chemical properties; A = atomic mass (sum of protons and neutrons), which differentiates isotopes).

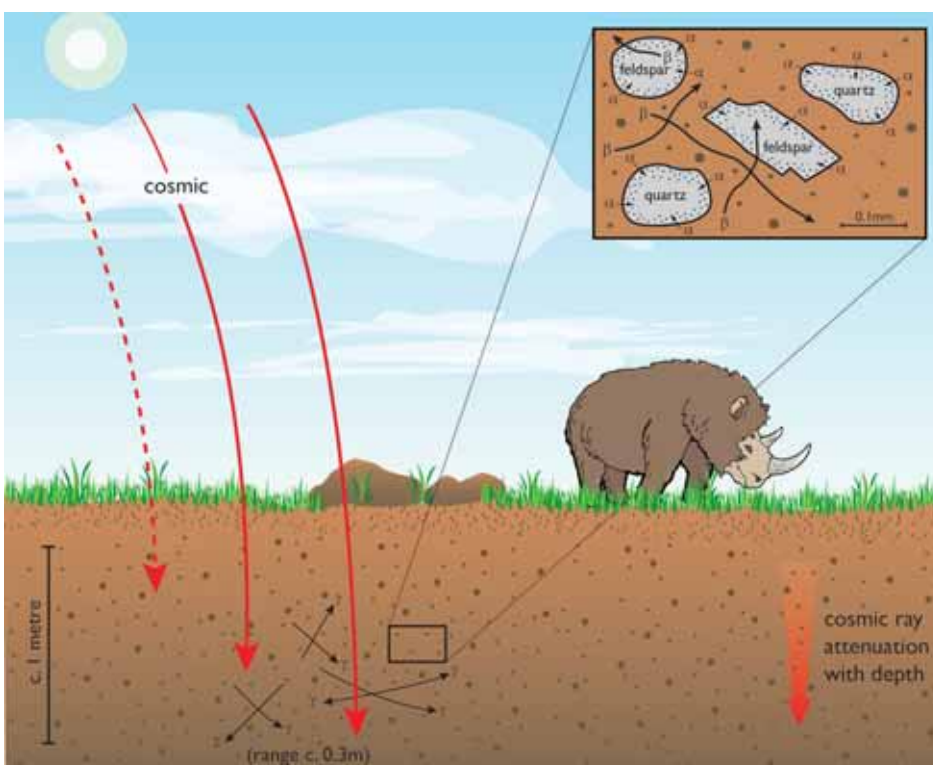


Fig 24 Environmental sources of radiation, showing ranges for alpha (α), beta (β) and gamma (γ) radiation and the decrease in cosmic rays with increasing depth below ground.

normally differentiate between the different uranium decay series (^{238}U , ^{235}U), nor do they normally measure the daughter products. Conventionally, calculation of the dose rate from 1ppm of U assumes that the ratio of the concentrations of ^{238}U to ^{235}U is about 400, and that all of the decay products are in secular equilibrium with their parent isotopes.

Secular equilibrium is the term used to describe a decay series (eg Fig 23) when the activities of each of the daughter products are the same. For luminescence dating this is commonly assumed to be the case, but in certain geochemical situations, this assumption may not hold (Olley *et al* 1996). For example, where water has percolated through sediment, this can selectively remove uranium (eg ^{238}U and ^{234}U) while leaving more immobile daughter isotopes such as thorium (eg ^{230}Th) behind. In seasonally wetted environments – such as marshes – where reducing conditions occur; radium (Ra) mobility is of concern. Where specific parts of a decay chain are either lost or gained in these processes, the decay chain is described as being 'in disequilibrium', meaning that the dose rate will change through time, making calculation of the luminescence age complex. After some period of time the rate at which the various isotopes are formed and decay will come into balance, and at that stage the sample is said to be 'in equilibrium'. In practice, difficulties associated with disequilibrium can be avoided by selecting samples from geochemical environments in which uranium, which is highly soluble, and thorium, which is insoluble, are not moving differentially. In particular; sediments that are within 0.3m (the range of gamma rays) of peat or other highly organic sediments should be avoided. Iron-stained sediments, especially where there is evidence for significant movement of groundwater; and samples with evidence for precipitation of calcite should also be avoided. However; when working in limestone caves such problems may be ubiquitous. Where disequilibrium is suspected, high-resolution gamma spectrometry (an emission counting technique described below) may be appropriate.

4.2 Emission counting

Instead of measuring the concentration of U,Th and K and using these to calculate the dose rate, a more direct method is to count the alpha, beta and gamma radiation.

Alpha particles can be counted by using either a thick source alpha counter (TSAC) or alpha spectroscopy. Where disequilibrium is suspected in a sample (see discussion in section 4.1), alpha and gamma spectroscopy are the best ways of studying the U and Th decay series.

TSAC is able to measure the total number of alpha particles, and by exploiting the rapid decay of ^{220}Rn (radon) in the Th decay series, the relative proportion of the alpha particles that come from the U and Th decay series can be calculated separately.

Alpha spectroscopy is more complex analytically, requiring the sample to be dissolved; but by using this method the energy of each alpha particle can be measured, which is characteristic of the isotope undergoing decay. In the same way, the energy of gamma radiation emitted from a sample is characteristic of the isotope undergoing decay. High purity germanium (HpGe) detectors, kept at liquid nitrogen temperatures (-196°C), are able to measure this energy, and produce a spectrum for each sample (Fig 25).

Beta particles, unlike alpha particles and gamma rays, are emitted at a range of energies, and thus there is no point in spectroscopy. However, simply counting the beta particles is perfectly feasible, for example by using a Geiger-Müller based system, and this directly assesses the beta dose rate.

One radioactive element, radon (Rn), is a gas and thus there is the potential for it to diffuse out of sediments. Rn isotopes occur in the ^{232}Th , ^{235}U and ^{238}U decay chains, but they have different half-lives – ranging from 3.96s (^{219}Rn) to 3.83 days (^{222}Rn). The longer-lived isotope ^{222}Rn is part of the ^{238}U decay series (Fig 23), and because of its longevity there may be time for the gas to move through the pores between sediment grains and escape into the atmosphere. If this occurs, the daughter isotopes will be produced away from the sample, thus reducing the radiation dose rate and causing disequilibrium in the decay chain. This situation will not cause difficulties if emission counting methods are used, but could be an issue if only parent concentrations (eg U and Th) are measured using chemical methods. These methods enable alpha, beta and gamma radiation flux to be measured, and alpha and gamma spectroscopy enable assessment of the extent of equilibrium in the decay chains.

Owing to the distances they travel, laboratory measurements of the alpha and beta flux on a small (c 10g) sub-sample are likely to accurately measure the natural flux of the sample. However, gamma radiation travels up to 0.3m in sediments. Laboratory measurements, based on emission counting or chemical methods, will only accurately determine the gamma dose if the material around the sample is homogeneous over a scale of c 0.3m in all directions. In many situations this will not be the case and then *in*

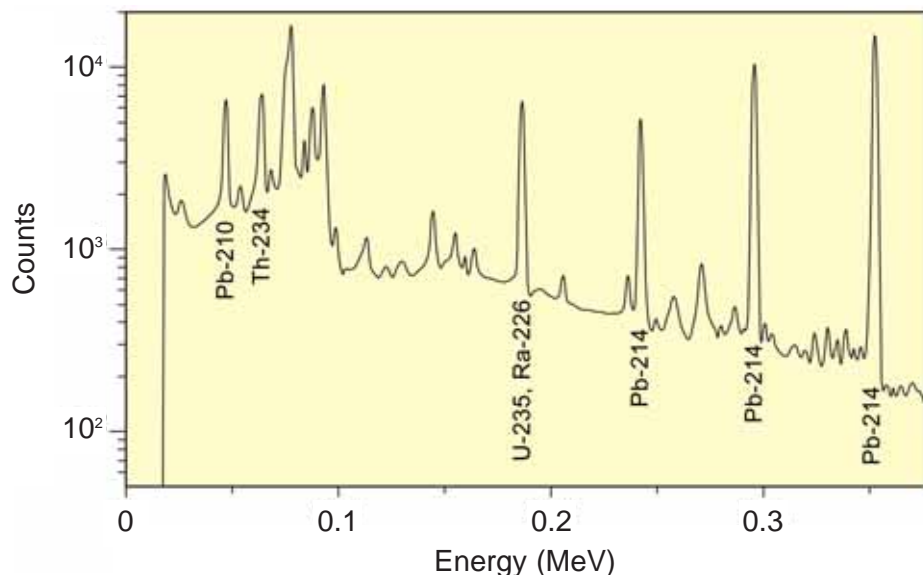


Fig 25 Part of a spectrum obtained from gamma spectroscopy; gamma rays emitted by specific isotopes have fixed energies; some of the more easily identifiable isotopes are shown (modified from Aitken 1985).

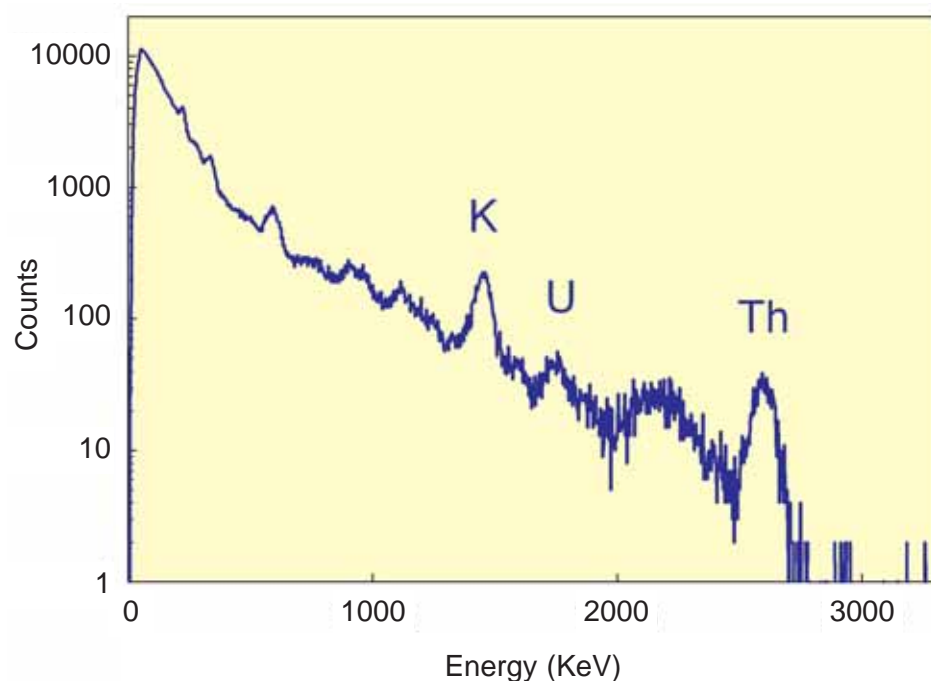


Fig 26 (above) Measurement was undertaken for one hour and produced the energy spectrum shown (peaks used to estimate K, U and Th concentrations are marked – the energy peak of 1460keV from K is particularly prominent). (left) A portable gamma spectrometer being used to make an *in situ* measurement of the gamma dose rate for a sample collected from a buried surface preserved within Silbury Hill, Wiltshire.

situ measurements need to be made.

4.3 *In situ* measurements

There are two approaches. One is to use artificial phosphors that are sensitive to radiation – for example, specially treated calcium fluoride or aluminium oxide. These can be buried – at least 0.3m deep in order to get a complete gamma field – at a site for between a few months and a year (or more), then recovered and their luminescence measured. This approach has the advantage that only small quantities of artificial phosphor need to be buried, in capsules c 10mm in diameter; thus minimising disruption to the site. The disadvantage is the need to visit the site twice.

The second approach, and the one used more commonly, is to use a portable gamma spectrometer. In laboratory measurements HpGe crystals used in high-resolution gamma spectrometry must be at liquid nitrogen temperatures. As this is impracticable in the field, sodium iodide (NaI) crystals that can be operated at room temperature are used. The quality of the spectrum obtained from NaI crystals is much poorer than that from HpGe, and so it is not possible to detect the same number of isotopes. However, peaks in the spectra obtained from a NaI crystal can be assigned to the U and Th decay series, and to K (Fig 26). The advantage of such *in situ* measurements is that they accurately capture the gamma dose rate, even if there is heterogeneity in the radiation field. The drawback is that such measurements require the probe to be inserted into a position as close as possible to the exact location of sample collection. Additionally, most NaI crystals used in luminescence researches are c 50mm in diameter, requiring a hole at least this wide and 0.3m deep to ensure that a complete gamma field is measured. To obtain a complete spectrum such as that shown in Fig 26 requires approximately one hour per sample, although this will vary depending on how radioactive the site is. Smaller NaI crystals are available, but require commensurately longer counting times. More rapid measurements can be made by integrating the area under the curve in Fig 26; however, such measurements will yield only the total gamma dose rate, rather than information about the individual U, Th and K concentrations.

4.4 The impact of water content

Except *in situ* measurements, all the methods described above are made on dried sub-samples. Water between mineral grains in the environment absorbs some of the radiation, meaning that only a proportion of the radiation emitted by the U, Th and K is absorbed by the mineral grains making up the

sample. The larger the amount of water, the less radiation is absorbed by the minerals. Calculations can compensate for this effect, but they require an estimate of the water content throughout the burial period. This is a difficult parameter to estimate, but some constraints can be put upon the value.

The upper limit can be determined by measuring the saturation water content (typically no more than 20–30% for sandy sediments), and by making measurements of the modern-day water content. A reasonable range of values can then be estimated by the site researcher and luminescence researcher. The precise impact of water on dose rate, and hence the age, will vary from sample to sample, but typically, a 1% increase in water content will increase the calculated age by c 1%. Particularly for sediments, uncertainty in the water content is commonly one of the largest sources of uncertainty in the final age estimate derived using luminescence, so careful consideration of this is essential. For caves, dunes and lakes, the uncertainty is likely to be fairly small, while for alluvial deposits it may be larger.

Samples with a high proportion of peat should be avoided because of the potential for disequilibrium (see section 4.1). However, if analysed, particular care is needed in estimating the water content, as it may have changed through time due to compression and dewatering. Additionally, organic matter absorbs radiation differently from water; and thus specific attenuation factors need to be used in dose rate calculations.

4.5 Cosmic ray contribution

Cosmic rays are an additional source of radiation. The cosmic ray dose rate depends primarily on three parameters: latitude, altitude and the thickness of any sediments or other materials that overlie the sample. The shielding provided by the Earth's magnetic field varies with latitude, and thus the dose rate increases closer to the poles, where the shielding is least. However, this effect is normally only significant for latitudes greater than c 60°N or S. Shielding from the atmosphere depends on its thickness; thus dose rate increases with altitude.

Standard calculations for the cosmic dose rate are appropriate for samples taken close to sea level. In practice the altitude normally only needs to be considered at heights > c 500m above sea level. The most significant factor is the thickness of any sediment, rock (especially in cave sites) or built structures overlying the sample. For sites near sea level in mid-latitudes, at a depth of 0.3m the cosmic dose rate is c 0.2Gy/ka, while at 10m it falls to c 0.07Gy/ka.

The effect of overlying sediments or buildings can be calculated accurately when the thickness of this shielding is known. In many cases samples might have been buried relatively rapidly following deposition, and the thickness of the overburden has remained effectively constant; but this will not always be the case. For many samples, the cosmic ray dose is a small proportion of the total dose. Typical silts and sands have combined beta and gamma dose rates of c 2 to 3Gy/ka, and the cosmic dose rate will be c 0.1 to 0.2Gy/ka – less than 10% of the total.

Calculating the cosmic dose rate is straightforward for most samples. As it constitutes only about 5% to 10% of the total dose it makes any uncertainty in the value relatively unimportant. However, where concentrations of U, Th and K are low, cosmic dose rate becomes proportionately more important. In carbonate-rich sediments – as are commonly found in East Anglia and parts of south-east England – beta and gamma dose rates may be closer to 1Gy/ka or less. For these samples cosmic dose rate may be 20% or more of the total.

The situation is more complex if the thickness of the overburden has changed dramatically through time – for example when sediments have accumulated steadily through time, increasingly shielding the sample from cosmic rays. If there is some indication of the timing of these changes, the impact can be modelled.

5 Limits of luminescence dating

5.1 Suitability of material

The quality of luminescence dating results are crucially dependent on the suitability of the samples, and centrally, whether a useful luminescence signal can be obtained.

Predicting where such samples will be encountered is not always simple, and this is why a pilot study can be important. In general, quartz from heated materials tends to give brighter signals, so pottery, burnt flints and bricks generally give bright signals. In unheated sediments, a major control on the brightness seems to be the initial geological origin of the quartz. General observations indicate that quartz derived from granites and from hydrothermal veins are dim. Once the grains have been through many cycles of reworking (eg to form a sandstone) there is a higher percentage of bright grains.

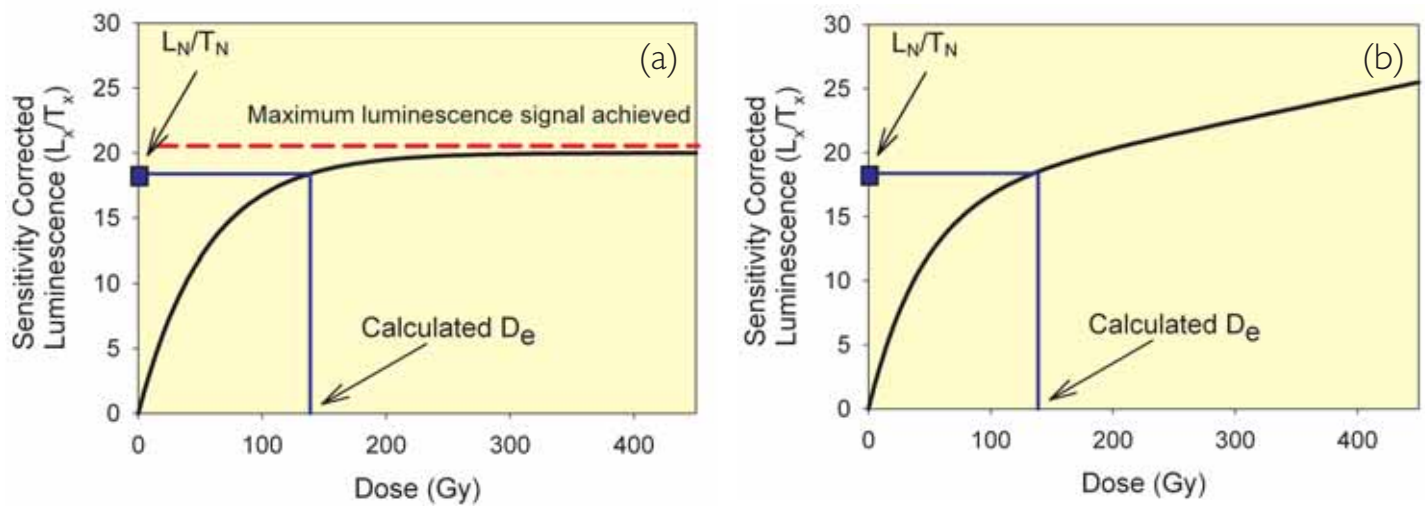


Fig 27 (a) At low doses the luminescence signal grows almost in a straight line, but at higher doses it grows more slowly. Ultimately the signal stops growing, when the sample is said to be saturated. This limits the age range to which the method can be applied. For this sample, it is effectively saturated at c 200Gy; if the dose rate were 2Gy/ka, the dating limit would be < 100ka. (b) Other aliquots or samples show patterns where the signal begins to saturate, then continues to grow linearly. The reliability of using this additional growth at high doses is currently unknown.

5.2 Resetting of the trapped electron population

In all luminescence dating, the event being dated is the last time that the relevant trapped electron population in the crystal (Figs 2 and 4) was reduced to a low level. Thus the archaeological value of the age obtained will be related to whether that resetting event coincided with the archaeological event of interest, and the completeness of that resetting event.

For heated materials, the crucial issue is whether the sample was heated to a high enough temperature, and for a long enough period of time, for the trapped electron population to be removed. The plateau test (Fig 22) undertaken during TL measurements can be a useful indicator; and further confidence can be given if replicate samples yield consistent results.

For unheated samples, the critical factors are the duration and intensity of the light to which they were exposed at deposition. Inadequate exposure to daylight at deposition leaves a residual population of trapped electrons (leaving the sample incompletely bleached). If allowance for this is not made, the age of the sample will be overestimated.

Methods to explicitly test this for each sample are described in section 3.

5.3 Upper age limits

The range of ages over which luminescence dating is applicable varies from one site to another; depending on the nature of the luminescence signal and the dose rate from the environment. The upper age limit is normally controlled by saturation of the luminescence signal. Fig 27a shows that the

luminescence signal, whether OSL or TL, increases as the size of the prior radiation dose increases. At first, this increase with dose is almost linear; but at some stage the traps within the crystal where electrons can be stored become full. As this happens, the luminescence signal grows more slowly, until all the traps become full, whereupon the luminescence signal ceases to increase despite continued exposure to radiation. This is known as saturation. This saturation imposes an upper limit to luminescence dating.

The dose at which the sample saturates varies from one sample to another; as does the rate at which it receives radiation. Thus it is impossible to give a precise upper limit to the age that can be obtained; instead the limit will be the maximum value of D_e that can be measured.

It is for this reason that reports on luminescence ages should always contain an example of the dose response curve obtained, as this indicates how close the sample is to saturation. The OSL signal from quartz normally saturates at approximately 150–300Gy (although this range also varies between samples); and with typical dose rates of 2Gy/ka this gives an upper limit of 75–150ka.

It should also be noted that as samples approach saturation (c 200Gy in Fig 27a), errors in the D_e value increase, for two reasons: because as the dose response curve flattens, small uncertainties in the value of the natural signal being projected have a larger impact on D_e values (visible in the asymmetric error given on individual D_e values); and, more difficult to assess, because of the impact of systematic uncertainties in the measurement of the dose response curve – thus ages obtained

with D_e values close to the limit of saturation should be regarded with additional caution. At present there is no consensus on how close to the limit of saturation one can reliably go. Wintle and Murray (2006) suggest that it is only prudent to work in the range where the natural signal (L_N/T_N) is 85% or less of the maximum luminescence signal obtainable. In Fig 27a the maximum signal on the y axis is 20, while the natural signal is 18. Thus the natural is 90% of the maximum value and is beyond the range suggested by Wintle and Murray (2006). However, the reality is often more complex, for two reasons. First, the dose response curve shown in Fig 27a is for a single aliquot, and it has been commonly observed that the behaviours of different aliquots of a single sample are different at high doses. Thus, as higher D_e values are approached, some aliquots fall below the 85% limit suggested, while others do not. To discard only those aliquots that fail this test would lead to systematic bias. So should any sample in which one aliquot fails this test be rejected? Secondly, the dose response curve in Fig 27a increases to a maximum value (c 20 on the y axis), and does not increase further. This response is what would be expected if a single type of trap in the mineral were being filled, and is fitted with a mathematical expression known as a saturating exponential.

However, it is not uncommon for some aliquots, and some samples, to have dose response curves where the luminescence signal does not reach a maximum value, but instead seem to consist of two signals – one of which saturates, while the other appears to continue to grow to higher doses (Fig 27b). Here the criterion suggested by Wintle and Murray (2006) cannot easily be applied as the sample appears not to saturate.

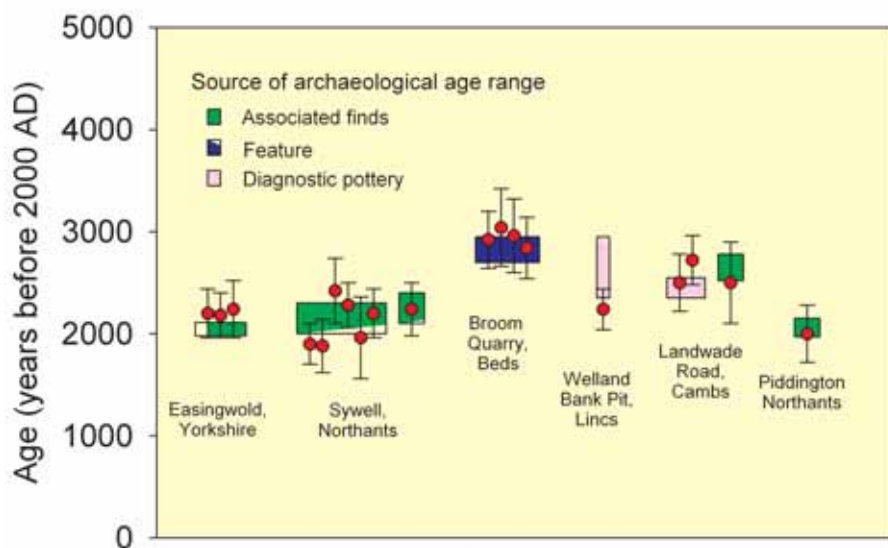


Fig 28 Comparison of TL ages on pottery and its archaeological age assessed on form and surface decoration (based on Barnett 2000); luminescence ages (red), with error bars (1σ , including random and systematic errors).

Table 1 Luminescence ages typically have errors between 5 and 10%. Illustrated here are examples of a 5% error, expressed in years (annum, a; or kiloannum, ka)

luminescence age (years before AD 2000)	central date	5% error ($\pm 1\sigma$)	calendrical bandwidth (68% confidence)	calendrical bandwidth (95% confidence)
42,000 $\pm 2100a$	40000 BC	2,100 years	42100–37900 BC	44200–35800 BC
22,000 $\pm 1100a$	20000 BC	1,100 years	21100–18900 BC	22200–17800 BC
12,000 $\pm 600a$	10000 BC	600 years	10600–9400 BC	11200–8800 BC
5,500 $\pm 275a$	3500 BC	275 years	3775–3225 BC	4050–2950 BC
4,500 $\pm 225a$	2500 BC	225 years	2725–2275 BC	2950–2050 BC
3,800 $\pm 190a$	1800 BC	190 years	1990–1610 BC	2180–1420 BC
2,600 $\pm 130a$	600 BC	130 years	730–470 BC	860–340 BC
1,900 $\pm 95a$	100 AD	95 years	AD 5–195	90 BC–290 AD
1,000 $\pm 50a$	1000 AD	50 years	AD 950–1050	AD 900–1100
300 $\pm 15a$	1700 AD	15 years	AD 1685–1715	AD 1670–1730
100 $\pm 5a$	1900 AD	5 years	AD 1895–1905	AD 1890–1910

However, the cause of the continued growth of the luminescence signal at high doses is at present poorly understood, and it is not known whether this can reliably be used in dating. Current results are equivocal. There are examples in the literature where old ages were obtained using dose response curves with forms such as that in Fig 27b, and which agreed well with independent age control (eg TL ages of Huntley *et al* 1993). A useful analogy for the present situation is the application of radiocarbon dating to old samples in the 1980s and 1990s. Radiocarbon ages ranging from 35ka to 40ka were obtained by laboratories for a number of samples, including ones that were key to archaeology (Roberts *et al* 1994), and these results yielded analytical errors suggesting that they had relatively small uncertainties. However, the introduction of trace amounts of young carbon – either during sampling or in sample preparation – had caused some of these ages to be erroneous. In the light of subsequent

improvements in techniques, it has become clear that not all the radiocarbon ages in the range 35–40 ka were wrong; but equally not all were correct. It is likely that a similar situation prevails in the luminescence analysis of old samples.

5.4 Lower age limits

The lower age limit is also difficult to define. This is controlled by two factors. The first is how well the luminescence signal was reset at the time of the event being dated. The second is the luminescence sensitivity of the mineral being studied. For young samples, D_e is likely to be small, and so the luminescence signal may be weak. Recent OSL studies show that in favourable conditions – for example in application to coastal dunes – ages in the last few centuries can be achieved (eg Bailey *et al* 2001; Sommersville *et al* 2003); and for heated materials, such as bricks, results for the last few centuries can be obtained (Bailiff 2007). For young samples it is particularly important to

undertake luminescence measurements to determine the most appropriate preheat treatment (see section 3.1.2, Fig 15). This is because thermal transfer can erroneously increase the apparent age of sediments if too high a preheat temperature is used.

5.5 Accuracy and precision

The accuracy of luminescence ages depends on a number of parameters. As discussed in Part B, it is essential that as much measurement information as possible is included in any report or publication dealing with luminescence ages. A number of compilations of ages measured on samples of known age have been produced. For sediments dated using quartz OSL measurements and the SAR protocol, a comprehensive review was given by Murray and Olley (2002), demonstrating good concordance with independent age estimates for the range from a few decades to more than 200ka. Barnett (2000) undertook a programme of TL dating of pottery from later prehistoric Britain that had been used to define the typological framework for that period. She found that where diagnostic form and surface decoration were present, the agreement between the luminescence ages on the pottery and the ages from independent methods was excellent (Fig 28). A recent comparison of luminescence ages measured on bricks with independent architectural evidence (ranging from AD 1390 to 1740) showed that the mean difference between the luminescence age and the assigned ages was 5 ± 10 years (Bailiff 2007; see Part C, section 14).

Limitations on the precision of luminescence ages have been described (section 5.3 and 5.4 above). When uncertainties in the measurement of the dose rate – often dominated by issues of water content (see section 4.4) – and D_e are combined, errors quoted on luminescence ages normally range from 5 to 10%, including both random and systematic sources of error. Luminescence ages are normally quoted at one standard deviation; that is, at the 68% confidence interval. Luminescence ages are obtained directly in calendar years, and thus do not require further adjustment. Table 1 illustrates the calendar ranges for luminescence ages with a 5% error. The example is shown for ages determined in AD 2000. There is no commonly accepted datum for luminescence ages, and so ages are expressed in years before the date of measurement. The term BP (before present) should never be applied to luminescence ages as BP has specific meaning relevant only to radiocarbon ages. These issues and how to quote luminescence

ages are discussed in section 10.

Part B Practicalities

6 Project management under MoRPHE

The potential for luminescence dating to contribute to a project should be identified as early as possible. Under *Management of Research Projects in the Historic Environment* (MoRPHE) guidelines (Lee 2006), it should form part of the project design document. Advice may be required on whether the archaeological question under consideration can be resolved by luminescence dating and, if so, whether the available samples are suitable for analysis. This advice should be sought in the first instance from an English Heritage Regional Science Advisor (see Appendix 1). More technical advice may be obtained from the English Heritage Scientific Dating Team or from the collaborating luminescence laboratory (see Appendix 2).

Where possible, a pilot project to assess the suitability of the material is advisable. This may be complicated by the turn-around time of laboratories (typically six months), but if this fits within the framework of the project then it is a valuable exercise. It is often difficult, even for someone with many years experience of luminescence, to be confident that samples from a specific site will be suitable for analysis unless a pilot study is carried out.

Luminescence dating requires the assessment of many variables, and while many of these do not require specific site information, there are some issues that do require this. Close collaboration between the project director and the luminescence specialist throughout the process will maximise the probability of obtaining high-quality chronological information. It is likely that personnel from a luminescence laboratory will want to collect their own samples, and that they will want to make a measurement of the gamma radiation dose rate in the field. At the same time they will want to assess the water content relevant to the sample, and with the project director consider whether this is likely to have changed through the period being dated.

The sampling approach, the materials to be sampled, the likely number of samples needed and details of how sampling is to be undertaken should be discussed with the luminescence specialist and the field team as part of assessing the dating potential.

The results obtained from luminescence dating must be archived. A large number of measurements are undertaken as part of

obtaining a luminescence age, and many different approaches may be taken depending on the precise nature of the material being dated and the methods used. The report should give this information in full, both to enable the excavator to judge the validity of the results, and for future researchers to assess the results in the light of subsequent developments in knowledge. The specifications for reports are laid out in section 9.

7 Sampling considerations

The most important part of using luminescence dating effectively is designing an appropriate sampling strategy. This will be constrained by the suitability of material available at the site, but ultimately it is essential that the strategy is driven by a clear idea of the archaeological question that is being asked. Where possible, luminescence dating should be used in conjunction with other chronological information: other dating methods (eg radiocarbon), documentary sources and stylistic information from artefacts. Samples for luminescence dating can broadly be classified into two groups: those including samples that were heated in antiquity and those made up of sediments whose grains were exposed to daylight during transport or deposition.

Samples that were heated in antiquity, leading to removal of trapped electrons in the mineral grains, date the heating event. This includes pottery, burnt flints and bricks. For all heated samples, a crucial issue is whether the mineral grains were heated to a sufficiently high temperature for their luminescence signal to be reset. Generally, a sample must reach c 300–400°C, for a duration dependent on its size.

Samples from sediments whose grains were exposed to daylight during transport or deposition date the time of transport or deposition. The most suitable sediments are those that were exposed to the most daylight during transportation, including wind-blown sands and silts. The optically stimulated luminescence (OSL) signal from minerals is more rapidly reset by daylight than is the thermoluminescence (TL) signal (Fig 8). OSL measurements have made it feasible to look at fluvial and colluvial sediments as well, but care needs to be taken in these cases to assess whether they were exposed to sufficient daylight at deposition to reset the luminescence signal being measured.

For all samples, care should be taken to avoid exposure to any strong source of radioactivity. This includes X-rays (as from XRF core scanners or hand-held XRF devices), as these

will increase the trapped electron population and make the sample appear older than it really is. Fortunately, current evidence is that X-ray systems used for security checks on commercial air flights or in customs for postal deliveries result in only a small dose. However, if the use of such devices has already occurred, or is likely, it should be discussed with a laboratory, as it may become significant for the analysis of young samples.

Where feasible, it may be prudent to collect duplicate samples: one for analysis by the luminescence laboratory and one for archiving. This is particularly useful in rescue archaeology, where sites are destroyed, or where sites are likely to become inaccessible due to building. Archived samples should be kept in cool, dry conditions, and their existence should be noted in the site report. If they are rich in organic matter they may require cold storage to prevent degradation.

7.1 Sampling strategy

A number of questions can be used to help design a sampling strategy. The first decision is whether luminescence dating is appropriate for a specific site:

- Are there materials at the site suitable for luminescence dating?
- Would a luminescence age for these samples be clearly related to the archaeological event that is of interest? There may be tension between selecting the sample that is closest to the event of interest and the optimal site for sample

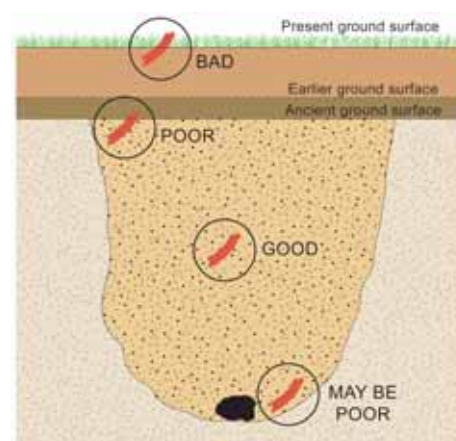


Fig 29 Selecting the optimal location for sampling is important. The best position is from the centre of the infill, where the material surrounding the sample is homogeneous. One consideration is the complexity of the gamma dose rate environment – dose rate will have changed through time near former ground surfaces, causing difficulties in assessment. The rock at the base of this pit may have a radionuclide concentration different from its surroundings, so calculating the gamma dose rate for samples collected near it may also be difficult (redrawn from Aitken 1990).

collection (eg Fig 29).

- Are the samples likely to be in the age range of luminescence dating for that material? (see sections 5.3 and 5.4)
- Will the precision of a luminescence age (typically 5–10% at $\pm 1\sigma$) be sufficient to answer the archaeological question and distinguish between different possible answers? (see Table 1)

If it is concluded that luminescence might be applicable, then the details of sampling should be considered:

- How many samples are required to answer the archaeological question(s)? Single ages from sites have limited value; multiple age determinations generally yield much more information.
- Are there clearly defined stratigraphic and/or chronological relationships between samples? This may significantly enhance the value of the ages. For example, Bayesian modelling of the luminescence ages may be possible to refine the dating of the site.

The details of these decisions will vary from one site to another; and a good sampling strategy is probably best designed in discussions between the project manager and the luminescence specialist. Regional Science Advisors and the English Heritage Scientific Dating Team (see Appendix 1) also have experience that may be of great value in this process. Some examples of different sampling strategies are given in Part C.

7.2 Heated samples

Pottery generally comprises a mixture of clays and coarser-grained materials used to temper the pot. Providing that the pot was fired to a sufficiently high temperature when it was manufactured, luminescence measurements can be made on either silt particles or, more commonly, sand-sized quartz inclusions in the temper. The dose rate to the sample is derived from both the pot itself and the surrounding soil.

The outer 2mm of the pot is removed in the laboratory so that the beta dose rate originates entirely from within the sample: thick sherds are needed for this, and most laboratories prefer ≥ 10 mm thick and 30mm across. Ideally, several sherds should be collected from each context, including a variety of fabrics to provide the laboratory with a range of material to work with. Because the surface is removed in the laboratory, the sherd does not have to be protected from light; thus it can be washed and recorded in the normal way prior to submission. As with all luminescence samples, the best samples are

from at least 0.3m from any major change in context, so that the gamma dose rate is homogeneous (Fig 29).

A sample of the soil surrounding the sample should also be collected, for measuring the gamma dose rate and water content. The soil sample need not be kept in the dark, but should be sealed in an airtight plastic bag as soon as it is excavated so that the water content is preserved. Sites should be avoided where the gamma dose rate is likely to have changed through time, such as near current or former land surfaces. Where samples must be collected near a context boundary (eg between an infill and the undisturbed surroundings) *in situ* measurements of the gamma dose rate is essential (eg using a portable gamma spectrometer).

Burnt flints can also provide thermoluminescence dates (Valladas 1992; Richter and Krbetschek 2006). Although flint is not common, at sites where it has been used for tool making it provides good material for dating. However, flints can only be dated if they were heated during antiquity, for example when pieces accidentally fell into a fire or if the flint was deliberately heated to improve its knapping properties. It is not always possible in the field to identify whether flints have been burnt, but surface features such as conchoidal fractures and colour may help to select pieces most likely to have been burnt; therefore when sampling, it is advisable to collect as many flints as possible.

Flint is a form of silica similar to quartz and has excellent luminescence properties: it gives a bright TL signal but no OSL signal. Its radioactivity is normally low, so the major contribution to the dose rate is the gamma dose rate from the surrounding soil. As with pottery, it is therefore essential to remove the outer 2mm of a sample in the laboratory using a diamond saw, to remove the beta contribution from the surrounding soil (Fig 30). Consequently, to get the required c 1g sample the initial sample needs to be c 10g. Such samples are, unfortunately, quite rare. Additionally, it is important to assess whether the sample has been heated throughout its mass to a sufficiently high temperature (c 300–400°C) to remove any pre-existing trapped electrons. Therefore for larger samples, the duration of heating would have to have been longer.

Thermoluminescence measurements are still routinely undertaken on flints, dating the last heating of the sample in antiquity. Additionally, by comparing the TL signal from the natural signal to that from an artificial irradiation, one

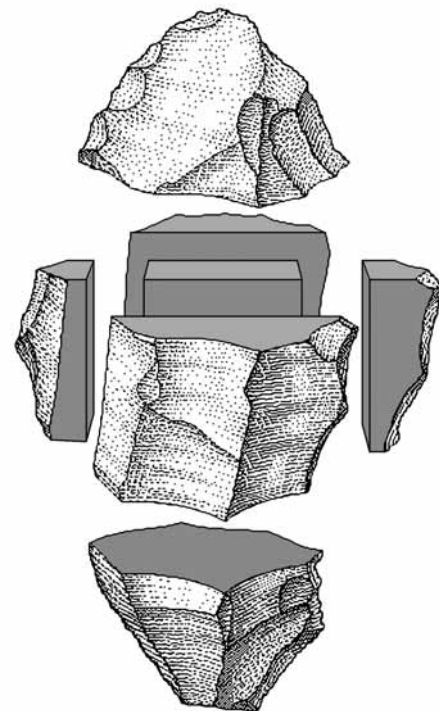


Fig 30 Removing the outer surfaces of a burnt flint with a diamond saw – the inner core is crushed and the material used to measure luminescence (diagram courtesy of Dr N Debenham).

can measure a TL plateau (Fig 22). As for pottery, accurate assessment of the gamma dose rate is essential – requiring careful selection of the sample locations – from either *in situ* measurements or collection of a sample of surrounding soil (Fig 29).

Fired clay bricks have been a common component in building construction over the last two millennia, and potentially provide suitable material for luminescence dating. As with pottery, either silt-sized grains in the brick matrix or coarser quartz sand inclusions from the temper can be analysed. More recent work has focussed on quartz sand inclusions, using OSL measurements, as they provide greater sensitivity than TL (Bailiff 2007). Samples can be taken from a brick using a diamond-tipped core drill. Bailiff (2007) used a 50mm diameter drill, and sampled to depths of 100mm. This provided an adequate length after discarding the outer 1–2mm of the core affected by the drilling.

Accurate assessment of the gamma dose rate is essential, as the gamma field may be complex. This can be done *in situ* using a gamma spectrometer or capsule dosimeter (see section 4.3).

7.3 Sediments (unheated samples)

In theory almost any sediment can be dated using luminescence, but three major

constraints reduce the applicability. The first is the physical nature of the sediment. Luminescence analysis requires the separation of quartz (or sometimes feldspar) grains. Quartz is the most ubiquitous mineral at the Earth's surface, but it is not common in some areas – for instance in areas where the underlying geology is carbonate rich (eg limestone or chalk), or clay rich (eg slates). The second constraint relates to measuring the sample dose rate. Luminescence analysis is undertaken either on silt grains (typically 4–11 µm diam) or on sand (90–300 µm diam). Therefore, clay or sediments dominated by gravels and larger grain sizes are unlikely to be suitable.

The third constraint is whether the mineral grains were likely to have been exposed to daylight at the time of deposition, or immediately before this. The most suitable sedimentary environments are wind-blown sands or silts (eg coastal dunes or loess). In recent years, measurement of the OSL signal, which is reset by exposure to daylight much more rapidly than TL (Fig 8), has made it possible to date other sediment types. Fluvial sediments, and sediments derived by slope wash, may be suitable. However, in these cases it would be important to make replicate measurements on samples to assess the

reliability of the ages: the approach (see section 3.3) relies on reducing the number of grains in each aliquot so that variations in the D_e from one sub-sample to another become visible, and thus it is only applicable to sand grains, as it is impossible to reduce the number of grains sufficiently and still obtain sufficient signals from silt.

Dates for the construction of standing stones and other prehistoric monuments have been attempted by measuring the luminescence of the last exposure of sediments underlying the stones or features (eg Rink and Bartoll 2005). Such an approach requires careful sampling from underneath the stones or structures, and it assumes that the ground surface was bleached prior to construction.

When taking sediment samples it is important not to expose them to daylight. The best method for achieving this depends on the nature of the sediment. The simplest approach is to use an opaque sample tube – plastic or metal, sufficiently thick-walled to exclude daylight, and mechanically strong enough to be pushed or hammered into the section. The size of the sample required, and hence the tube, will vary depending on the nature of the sediment. Typical sample tubes are c 70mm in diameter and 0.2–0.3m long. It may be possible

to use smaller sample tubes, but this should be discussed with the luminescence specialist before collection. Immediately before pushing the tube into the face of the section, a few millimetres of sediment should be removed from the surface, as these grains will have been exposed to daylight. After filling the tube with sediment, it should be wrapped in thick black plastic. Any spaces left in the tube should be packed with plastic bags to secure the sample in transit, sealed with tape and clearly labelled with a black marker (red marking is invisible in luminescence laboratory lighting conditions). Where sediment is too hard or stones in it prevent the use of a tube it may be necessary to excavate a sample shielded by a black tarpaulin to exclude daylight (Fig 31). The sampler can use a dim red light (eg a bicycle rear light) when collecting, then put the sample into an opaque black plastic bag. Black 'builders plastic' made into bags with a heat sealer, or bags used for X-ray plates by hospitals are suitable; but black bin liners are not sufficiently thick. Luminescence laboratories may be able to supply black bags.

The volume of sample will depend on the nature of the sediment, but a good guide is 500g. In some circumstances the material may be sufficiently hard to carve out an intact block sample using a trowel or similar implement. The block must be wrapped in black plastic, aluminium foil or other opaque material, and supported to prevent it from disintegrating in transport to the luminescence laboratory, for the outer surface of the block, which will have been exposed to daylight during its excavation, has to be removed. Thus the block must be large enough – typically c 100mm in each direction – to allow for this.

When it is especially difficult to exclude daylight during sampling or a block sample is impracticable, a last resort is to sample at night, using a red light.

Whatever sampling method is used, the hole can be used for making *in situ* gamma spectrometry measurements.

A number of options exist for sampling by coring below the ground surface. The best solution is to take samples using dedicated opaque core liners. Such core sections can be taken directly to the laboratory, where they can be sub-sampled under controlled laboratory lighting conditions. If this procedure is not possible, sub-sampling can be done on site under a black tarpaulin as soon as the core barrel reaches the surface.

In all cases, samples must be taken for measuring the water content. Sub-samples



Fig 31 Sampling fluvial gravels near Ripon, Yorkshire: a black plastic tarpaulin was used to exclude light and the sample was collected in a thick black plastic bag (photograph by G A T Duller).

collected for luminescence analysis, and completely sealed, can be used, or separate samples (c 20g) can be collected in air-tight plastic bags (which do not have to be kept dark).

7.4 Health and safety

Whatever type of sampling is being undertaken, it is important to assess health and safety risks. Such issues are unlikely to be unique to the sampler, but the difficulties of sampling without exposing samples to daylight may increase potential risks.

The greatest risks are probably those associated with sampling a section. The stability of the section should be checked, and where appropriate suitable shoring provided. A hard hat should be worn where appropriate. The dangers associated with sections are especially significant when using a tube to take a sample, as this will normally require hammering into the section, possibly dislodging material from the section. Equally, where the sampler is required to be underneath a tarpaulin, he or she will not be able to get away from the section rapidly should it fail, or any part of it become dislodged.

8 Laboratory considerations

Once submitted to a luminescence laboratory, a sample undergoes a complex sequence of preparation procedures and requires the measurement of a wide range of parameters. Preparation is carried out under subdued red lighting, similar to a photographic laboratory, whose light intensity and wavelength does not damage the trapped electron population. A number of chemical steps remove organic matter and carbonates, a specific grain size is separated out, and a specific mineral is isolated (Aitken 1985). The common approach for quartz uses density separation, which removes heavy minerals and some feldspars; and hydrofluoric acid, which etches away feldspars. These processes take time. Generally, results take six months, but this will depend on the laboratory workload and can be confirmed at the time of submission.

At the date of writing there is no formal accreditation scheme for luminescence laboratories or any recognised body to which they would be expected to belong; and no formal laboratory comparisons have been made. In the United Kingdom almost all luminescence laboratories are in universities or research institutes, and most are active in academic research and publish in archaeological or geographical journals. Appendix 2 is a list of laboratories and contact details. Their web sites have publication lists, including recent dating projects in which they

have been involved.

9 Reporting specifications

There are presently no specifications for the reporting of luminescence ages. A luminescence age calculation requires two parameters: D_e and dose rate. In turn, each parameter requires many specific measurements, and a variety of methods may be used for each. Lists of ages, or even summary D_e and dose rate values alone, provide insufficient information to assess whether the ages are meaningful.

Laboratory reports should include sufficient information

- to document the work that has been undertaken;
- enable the field project director or relevant person to assess the reliability of the ages produced and any difficulties that were encountered;
- and provide an archive of the methods used and the results obtained, so that the results can be assessed in the future in the light of subsequent developments in understanding of methods.

Reports from specialist luminescence laboratories should include information under the following headings.

9.1 Sample details

- A description of what was sampled and the precise context of the sample, giving as much information as possible. For multiple samples, stratigraphic or contextual relationships should be made clear. This is possible only if the luminescence specialist collects the samples, or has precise information from the excavator. Given the importance of estimating the sample water content and understanding the radiation context, having the specialist on site is of great value.
- For pottery and burnt flint, both the sample and the surrounding sediment should be described.
- A brief description of the collection method and, where appropriate, details of sample storage should be given.
- Photographs or detailed field drawings showing sampling locations should be made.
- The depth below the current ground surface should be recorded for the calculation of cosmic ray dose rate.

9.2 Luminescence measurements

- Describe the laboratory procedures used to prepare samples, indicating the grain size and mineralogy selected for luminescence measurements.

- Describe the equipment used to make luminescence measurements, specifying the filters used to detect the luminescence signal, and, in the case of OSL dating, the wavelengths and power used for optical stimulation.
- State the calibration of the artificial radiation source used during measurements. This should include the date of calibration and the external source used (eg National Physical Laboratory, or Risø National Laboratory), including details of the sources used (eg the ^{60}Co gamma source used to calibrate a ^{90}Sr beta source). Include the uncertainty associated with the calibration (usually 2–3%), and state that this uncertainty has been propagated into the total uncertainty on the luminescence age.
- State the method used for measuring D_e and the nature of the aliquots used for measurement (eg fine grains (4–11 μm diam) settled on a 9.8mm aluminium disk, or c 100 grains (180–211 μm diam) on a 9.8mm steel disk).
- Include graphs showing typical examples of the luminescence signal measured, any unusual behaviours observed and an example of a dose response curve, particularly for older samples that may be close to signal saturation.
- State the mathematical method used for deriving D_e (eg interpolation, or mathematical fitting of a dose response curve) and the method used to calculate the uncertainty on this D_e value (eg jackknifing, Monte Carlo methods, etc). Include a statement of the instrumental reproducibility (typically 1–2%) and how this has been included in the calculation of D_e .
- Note where samples are close to the limits of the method, either because they are very young or very old, and discuss the implications for the resulting ages.
- If multiple measurements of D_e have been made on each sample, state the number of replicates, including the number of aliquots rejected during analysis.
- Show an example of the distribution of D_e values; if there are complex distributions, show them all.
- State method used to combine multiple D_e estimates together in order to calculate the D_e value used for age calculation (eg finite mixture model, central age model, etc.).
- Describe details of quality control checks undertaken, and their results, to ensure the validity of the method of D_e determination. For example, using the SAR protocol on quartz routine checks should include a recycling ratio measurement, a dose recovery test, a zero-dose (recuperation) check and the results of varying the preheat

temperature.

9.3 Dose rate measurements

- Give a brief statement of the methods used for dose rate measurement, differentiating clearly between *in situ* measurements and laboratory based measurements.
- Give information about any outsourced dosimetry measurements (eg neutron activation analysis, or ICP-MS), including the laboratory used and the lower detection limit of the determination of each element.
- State whether an internal dose rate has been included; if so, state its value and how this was obtained. For quartz such a value is normally small ($< 0.05\text{Gy/ka}$), but may be larger if other minerals are used for measurement.
- State if any explicit measurements have been made to assess whether radioactive disequilibrium is present.
- Include a table of results, including water content value, grain size and separate enumeration of the components making up the total dose rate.
- Give a justification for the choice of water content used for dose rate calculation.
- Give a brief justification for the choice of overburden used for cosmic-ray dose rate calculation, and any adjustments made for site altitude and geomagnetic latitude.
- Where relevant, state how the alpha effectiveness (a-value) was assessed.

9.4 Age calculation

- State the dose rate conversion factors and the beta-dose attenuation factors employed.
- State the software (commercial or written by the laboratory itself) used to calculate the age.
- Make it clear if errors quoted for the age includes the total uncertainty (both systematic and random).
- Round up or down ages to an appropriate level: for ages < 1000 years round the age and its uncertainty to the nearest 5 years; for ages > 1000 years round to the nearest 10 years (eg $12891 \pm 768\text{a}$ suggests unfounded precision, and should be rounded to $12890 \pm 770\text{a}$, which could also be written $12.89 \pm 0.77\text{ka}$).
- State the datum used for calculating ages, normally the year of measurement.

9.5 Interpretation

The detail in this section will depend on the relationship between the luminescence specialist and the project. While many luminescence ages will be straightforward, there will be occasions where unexpected results are found. Such issues are most effectively dealt with through a close

collaboration and discussion between the luminescence specialist and the project director. Where possible, the outcomes of these discussions should be included in the final report so that the ages are put into their appropriate contexts.

Where available, the results should be compared with other lines of evidence.

9.6 Indexing

Luminescence reports should be indexed using one or more of the preferred terms adopted by the Forum on Information Standards in Heritage (FISH). These should describe the type of analysis and material analysed (Tables 2 and 3; NMR Archaeological Sciences Thesaurus), and the archaeological object or site being dated (MDA Object Type Thesaurus; NMR Thesaurus of Monument Types; http://thesaurus.english-heritage.org.uk/thesaurus.asp?thes_no=11). Any chemical methods of analysis used to determine the dose rate should also be indexed.

9.7 Non-technical summary

To aid readers in their use of the luminescence report it is helpful to provide a non-technical summary of the key findings of the study.

Table 2 Preferred terms used in the National Monuments Record (NMR) Archaeological Sciences Thesaurus for types of luminescence dating (http://thesaurus.english-heritage.org.uk/thesaurus.asp?thes_no=560). PDF versions of the thesaurus are available on request from the Data Standards Unit of English Heritage Luminescence Dating

- thermoluminescence (TL)
- optically stimulated luminescence (OSL)
- infrared stimulated luminescence (IRSL)

Table 3 Preferred terms used in the NMR Archaeological Sciences Thesaurus for the materials analysed (http://thesaurus.english-heritage.org.uk/thesaurus.asp?thes_no=144&thes%20name=MDA%20Object%20Type%20Thesaurus)

- brick
- burnt flint
- quartz
- feldspar
- zircon
- polymineral
- biogenic carbonate
- pottery
- geological sediment

10 Quoting ages and disseminating results

The following is a guide for quoting luminescence ages in reports and other publications.

Luminescence ages are calculated in years before the date of measurement. The unit of measurement is the annum (abbreviation 'a'). Thus an age of $110 \pm 10\text{a}$ measured in 1997 is dating the same event as one of $120 \pm 10\text{a}$ measured in 2007. There is no agreed convention about a datum year from which ages are measured, although some laboratories are starting to report ages in years before AD 2000. Thus it is important to make clear what datum is being used – normally this is the year of measurement.

The uncertainty in luminescence ages should always be quoted, indicating whether it represents one sigma (1σ) (68% confidence interval) or 2σ (95% confidence interval). Luminescence laboratories commonly quote errors at 1σ , but it should be noted that this error normally includes both random and systematic errors. All luminescence laboratories will assign unique sample codes (eg Aber29/RG6, X2451, GL03008, Dur05OSLqi-301-1), and these should be quoted with the age (eg '...the sample gave a luminescence age of $130 \pm 10\text{a}$ (Aber29/RG6) and demonstrates...').

Luminescence results presented as calendar dates should be given as age ranges. Thus a luminescence age of $1000 \pm 50\text{a}$ measured in 2000, could be expressed as a calendar range of AD 950–1050 (eg '...aeolian sand collected from level 3 gave a luminescence date of AD 950–1050 (X2451) ...'). It is also correct to quote ages as calendar dates with their associated errors. Thus a luminescence age of $955 \pm 12\text{a}$ measured in 2005 could be quoted as AD 1050 ± 12 .

Recording and disseminating the results from luminescence analysis must be done. Reports produced by the specialist luminescence laboratory form part of the final project archive, and a copy of them should be lodged with the local Historic Environment Record (HER). Their publication, either in a monograph resulting from the project, or in a separate research paper is also encouraged so the results have wider dissemination and undergo peer review.

Wherever luminescence ages are reported, it is crucial that information associated with generating those ages, is included, or at least is available. The report must include a brief

description of the analytical methods used, the materials analysed and summary tables, and give a reference to the original laboratory report. Where possible, the form of the luminescence signal measured (TL glow curve or OSL decay curve), the dose response curve, and the distribution of D_e values for a typical sample should be shown in figures. This is essential for readers to judge the quality of the age estimates.

Publication should normally involve personnel from the luminescence laboratory, to present the technical data involved in the measurements and to advise on interpretation of the results.

Summary tables should include:

- the laboratory code of the sample;
- the water content used in calculations of the dose rate;
- either the elemental concentrations of U, Th and K, or the alpha, beta and gamma dose rates;
- the cosmic dose rate and the total dose rate used for calculation of the age;
- D_e , and if appropriate the number of replicate measurements that were used in the calculation of the final D_e ;
- and the age of the sample.

Where possible, give the uncertainties in these values (eg water content $7 \pm 3\%$, or beta dose rate $1.44 \pm 0.07 \text{ Gy/ka}$). Where some of these values are the same for all samples, a footnote may help to simplify the table.

When it is not possible to include all the primary information, a reference should be made to a report or some widely accessible database where such information has been deposited. The Archaeology Data Service (ADS) (ads.ahds.ac.uk) or a specialist journal such as *Ancient TL* (www.aber.ac.uk/ancient-tl) may be an appropriate place to do this (see for example Barnett 1999).

Part C
Case Studies

11 Wat's Dyke, Gobowen

Wat's Dyke is an important feature in the historic landscape of the northern border region between Wales and England. When constructed, the ditch was up to 8m wide and 4m deep and ran for c 65km from south of Oswestry to the Dee Estuary. The dyke is thought to have been constructed sometime in the post-Roman period, but the exact date of its construction is poorly known and previous excavations have failed to uncover any significant artefacts to help determine its age.

Close to its southern limit, Wat's Dyke runs near Gobowen on the Welsh border. An area given planning permission for a housing development by Fletcher Homes included a clearly visible part of Wat's Dyke. Therefore Gifford Ltd undertook archaeological survey in 2006 before construction started. The primary objectives were to record the morphology of the dyke at this location and to retrieve samples for absolute dating.

Two trenches were excavated, revealing an in-filled ditch c 2.5m deep and 8m wide. Near the ditch there was a bank c 0.5m high and 5m wide. Excavation of two trenches across the ditch revealed a sequence of infilling sediments, but these had limited organic material suitable for radiocarbon dating (Fig 32).

The only sample directly associated with the ditch and suitable for radiocarbon dating was a charred twig found in the lowermost fill. It yielded a calibrated age of 1120–890 cal BC (at 2 standard deviations; SUERC-12826; 2855 ±40 BP), significantly older than the assumed post-Roman age.

It was decided to obtain an independent date using luminescence. A simple strategy for sampling would have been to collect a sample from as low down in the fill as possible, close to the undisturbed site. Such a sample would be difficult, if not impossible, to date using luminescence because there is often rapid infilling immediately after digging of this type of ditch, and the grains are unlikely to have been bleached. The materials into which the ditch was dug are fluvial sands and gravels which gave a luminescence age of 58.9 ±5.8ka (X2840). Moving away from the boundary and taking samples from areas that are more likely to have been deposited gradually, and to have

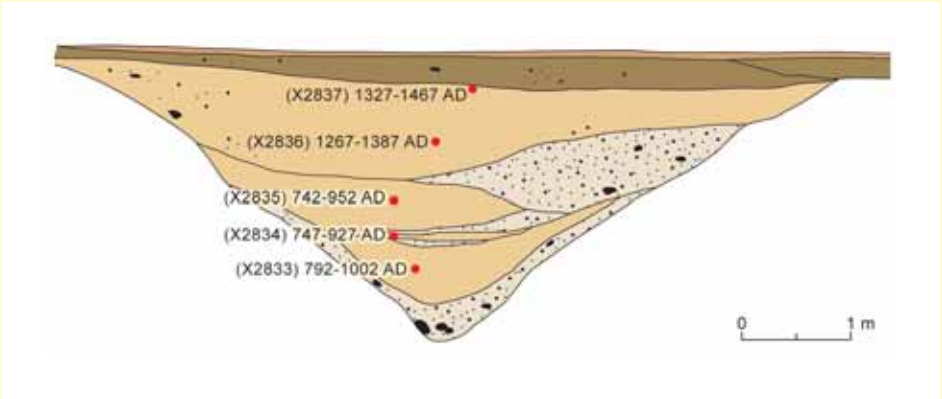


Fig 32 (above) Section drawing showing positions of the five samples, their laboratory codes and the ages obtained from them (range = 1σ or 68% confidence) (modified from Malim and Hayes, submitted). (below) Gamma spectrometry measurement *in situ* at Wat's Dyke, Gobowen – five samples (holes in excavated section) were collected for luminescence dating (photograph by J-L Schwenninger).

Table 4 Summary of luminescence data for samples from Wat's Dyke (from Malim and Hayes submitted): D_e for each sample is the average of 12 determinations					
lab code	depth	equivalent dose	dose rate	age (a)	date (AD)
	(m)	(D_e) (Gy)	(Gy/ka)	(before AD 2007)	(1σ range)
X2833	2.10	2.73 ±0.19	2.47 ±0.15	1110 ±105	792–1002
X2834	1.90	2.79 ±0.10	2.37 ±0.15	1170 ±90	747–927
X2835	1.55	2.72 ±0.16	2.34 ±0.15	1160 ±105	742–952
X2836	0.95	1.58 ±0.06	2.32 ±0.17	680 ±60	1267–1387
X2837	0.46	1.50 ±0.06	2.44 ±0.26	610 ±70	1327–1467
X2838	2.30	2.89 ±0.06	2.33 ±0.14	1240 ±85	682–852
X2839	0.40	2.88 ±0.08	2.59 ±0.29	1110 ±130	767–1027
X2840	0.60	185.5 ±10.8	3.15 ±0.24	58900 ±5800	

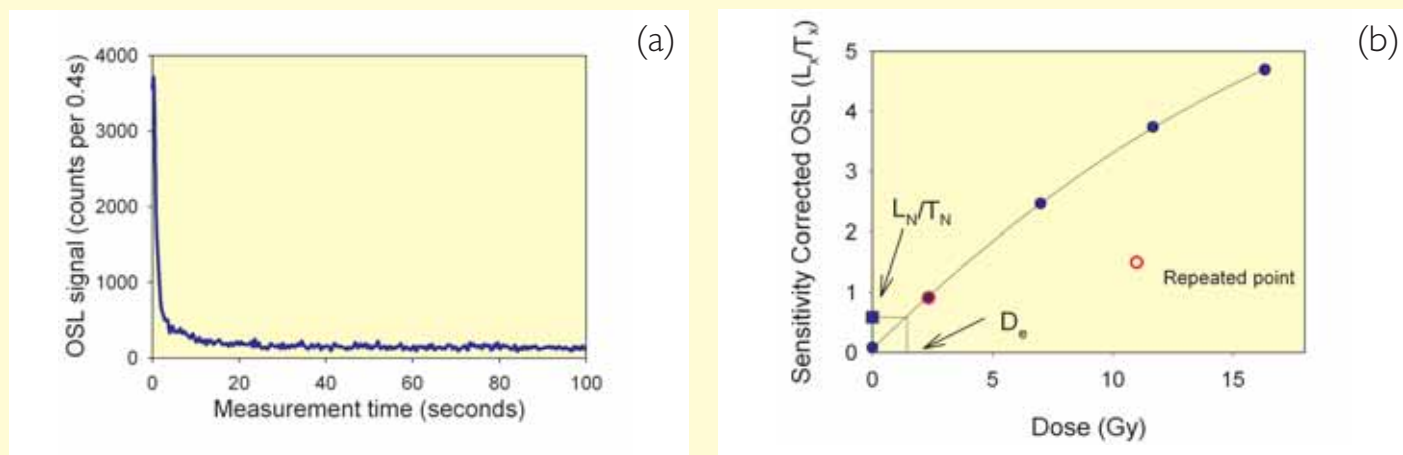


Fig 33 Luminescence data for a 4–5mm diam quartz aliquot from sample X2836, Wat's Dyke, Gobowen: (a) OSL decay curve for an aliquot measured using blue LEDs; (b) the dose response curve for the same aliquot, yielding a D_e of 1.45 ± 0.07 Gy – the regeneration dose at 2.5 Gy was measured twice (repeated measurement shown in red) and the recycling ratio of 1.00 ± 0.02 is within the acceptable range 0.9–1.1; (c) radial plot of D_e values for 12 aliquots of sample X2836 – only one D_e value falls outside the shaded band, suggesting that they all form a single population.

been bleached, would increase the accuracy of the luminescence age.

An additional means of confirming that samples were bleached before deposition is to take samples from contexts with clear stratigraphic relationships: ages consistent with these relationships gives reassurance of their accuracy and allows a Bayesian analysis of the results, giving additional information about the age model for the site.

At an early stage of excavation the project manager contacted the luminescence laboratory at the Research Laboratory for Archaeology and the History of Art (RLAHA) in Oxford and, in collaboration with the English Heritage Regional Science Advisor, they devised a sampling strategy to collect eight samples for luminescence dating: one from the fluvial sands and gravels of the underlying site geology and seven from the two trenches.

In one trench, a sample was collected at a depth of 2.30m and one at 0.40m; at the other

trench, five samples were collected at depths from 2.10m to 0.46m. In all cases, the samples were from contexts where evidence indicated slow infilling. Selection of samples from two trenches made it possible to assess the reproducibility between sites. Detailed sampling at one trench made possible a chronology for the infilling.

Samples for OSL dating were collected by hammering opaque black plastic tubes into the excavated section.

Sampling was done by RLAHA personnel in one day, at the same time making *in situ* gamma dose rate measurements with a gamma spectrometer (Fig 32), and taking samples for the measurement of sediment water content (which ranged from 11% to 22%, increasing with depth). The water content values were used to calculate the sample dose rate.

In the laboratory, quartz sand grains (180–255 μ m) were extracted and aliquots

4–5mm diam (c 100 grains per aliquot) were prepared. The OSL signal from these aliquots dropped rapidly in the first few seconds of measurement, then showed a slightly slower decrease from c 5s to c 20s (Fig 33a). D_e values were determined using the SAR procedure and the dose response curves show a smooth increase in OSL signal with dose. As normal, one of the regeneration doses (in this case 2.5 Gy) was given a second time, to assess the ability of the sample to recycle a dose, giving a recycling ratio of 1.00 ± 0.02 , well within the acceptable range (Fig 33b). Twelve replicate measurements of D_e were made on each sample. Those for X2836 are similar, and almost all fall within the shaded band in Fig 33c.

The ages for the two samples from near the base of the ditch were 1240 ± 85 a (X2838; expressed as years before 2007) and 1110 ± 105 a (X2833) (Table 4); 1σ errors on these ages give respective calendar dates of AD 682–852 (X2838) and AD 792–1002

(X2833). The dates of the sequence of five samples from one ditch show that within the uncertainties they agree with the stratigraphy, and thus document filling of the ditch between c AD 800 and the 14th century (Fig 32). This stratigraphic consistency supports the notion that the samples had been bleached before deposition. Further support for the luminescence chronology is provided by abraded sherds of a single wheel-thrown, lead-glazed vessel – discovered in an adjacent buried soil – dated to the 13th or 14th century and thus contemporary with infilling of the ditch by medieval ploughing.

The radiocarbon age of 1120–890 BC from the burnt twig recovered from the bottom of one trench cannot be easily reconciled with the luminescence ages, and it seems likely that this was introduced into the ditch fill from an earlier context when Wat's Dyke was in use.

The luminescence ages imply that infilling occurred in the early 9th century. Malim and Hayes (submitted) therefore postulated that at this site Wat's Dyke was constructed either in the reigns of Cenwulf and Ceolwulf (AD 723–796) or of Wiglaf during the 830s.

The excavation and analysis benefited from the enlightened approach of Fletcher Homes to the work undertaken prior to development of the site.

12 Dungeness Foreland, Sussex

Dungeness Foreland is the third largest coastal lowland in the United Kingdom, and has a rich Bronze Age to modern day archaeological record. The absence of archaeological finds predating the Bronze Age is consistent with the broad picture that had been built up of the formation of this feature during the last 4000 years. Drilling across the foreland showed that it comprised a series of gravel ridges (Fig 34), with an underlying sand body. Organic sediments had accumulated in the swales between the gravel ridges. The orientation of the gravel ridges, and earlier radiocarbon dating suggested that the feature grew from the south, towards the current tip of the foreland. However, the detailed chronology and pattern of deposition of the sands and gravels making up the foreland was unknown.

A project supported by an Aggregate Levy Sustainability Fund (ALSF) grant aimed to use OSL measurements to date the underlying sands, and radiocarbon to date the overlying organic sediments, thus providing the age and pattern of formation of the underlying sands, and bracketing the timing of the emplacement of the gravel ridges.

A contractor drilled twelve vibrocore boreholes to recover sands from below the gravel ridges. Their sites were chosen to form two transects, running approximately east–west and north–south. To provide stratigraphic control, 37 sand samples were collected: 3 samples from 11 of the boreholes, and 2 samples from the 12th borehole, plus 2 samples of the modern beach sand to assess whether they were completely bleached.

As the majority of samples were collected from several metres below the ground surface, it was not possible to measure gamma dose rates using *in situ* gamma spectrometry. Instead, laboratory measurements were made based on sub-samples of the material collected for dating. It was likely that the gravels and the sands would yield different gamma dose rates, so to ensure that the laboratory-based measurements gave accurate gamma dose values, sample locations were chosen that were at least 0.3m from the sand/gravel boundaries.

The water content of the samples was also considered. Most samples were collected from below modern sea level, and were expected to be saturated (c 25% water

content). Roberts and Plater (2005) also considered the potential for changes in water content associated with changes in sea level during last 5,000 years, and generated a simple model for this. They concluded that the effect would be negligible for all but two of the oldest samples.

The sand samples were collected in clear plastic liners (38mm diam), sealed in the field to prevent water loss and taken to the Aberystwyth Luminescence Research Laboratory.

As the core liner was transparent, the outer part of each core was exposed to daylight during collection. Under red-light in the laboratory a thin-walled, 20mm diam cylindrical sampling device was pushed up the length of each core liner to retrieve an inner sub-sample, unexposed to daylight. Quartz sand grains (150–80µm diam in this study) were isolated and the OSL signals measured using blue LEDs (giving a power of 17 mW/cm²).

Aliquots consisting of c 200 grains were measured from the majority of samples; larger aliquots of c 800 grains were used for the youngest samples, to increase the OSL signal and thus reduce the error on these samples (Roberts and Plater 2005).

Despite their young age, the sediments yielded relatively bright OSL signals (Fig 35); the signals dropped rapidly during the first 5s of OSL measurement as would be expected for quartz. The SAR procedure was used to construct a dose response curve for each aliquot and several tests were applied: a recycling test and a check to ensure that no feldspar contamination was present was applied to each aliquot, and a preheat test was undertaken for each sample (Fig 15). Only those aliquots that passed all of these tests were included for subsequent analysis.

A minimum of 21 aliquots were measured for each sample, and between 11 and 44 aliquots passed all the tests. The variation in the D_e values for each sample was analysed to see whether there was any indication of incomplete bleaching, but within each sample the spread was small (Fig 36), implying that the luminescence signal in the sediments had been bleached at deposition.

A sub-set of seven samples was also analysed using medium aliquots (c 200 grains) in order to assess whether any indication of incomplete bleaching was being masked by the larger aliquots, but no evidence was found for this.

Finally, a dose recovery test was applied to the samples. Together with the tests described above, favourable results gave confidence in the luminescence ages obtained.

The two samples collected from the modern beach gave ages of $40 \pm 40\text{a}$ (Aber73/BH-USS) and $15 \pm 15\text{a}$ (Aber73/BH-SSR) demonstrating that these beach sediments were completely

reset at deposition. The 35 OSL ages from the drill cores ranged from $430 \pm 20\text{a}$ to $5120 \pm 220\text{a}$ (expressed as years before 2000) (Table 5). By collecting three samples from each core, the veracity of the ages could be confirmed by observation of the stratigraphic consistency (Fig 37). Several samples give ages that are not in obvious order: eg in core 1, a value of $4700 \pm 200\text{a}$ (Aber73/BH-1/1) overlies

ages of 4070 ± 170 (Aber73/BH-1/2) and $5120 \pm 220\text{a}$ (Aber73/BH-1/3). However, these results are consistent within two standard deviations (ie $4700 - (2 \times 200) = 4300\text{a}$ and $4070 + (2 \times 170) = 4410\text{a}$) and suggest that within the precision of these ages, typically 5%, the two samples are very close in age. Bayesian analysis of this suite of data revealed only one sample whose OSL



Fig 34 Gravel ridges across Dungeness Foreland, looking north-east (photograph by H M Roberts).

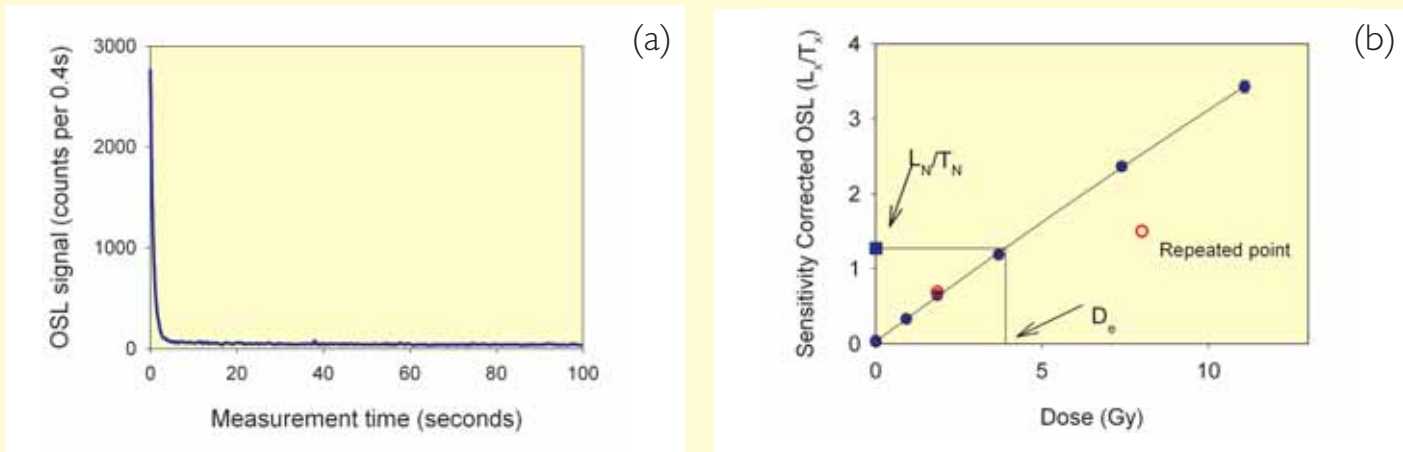
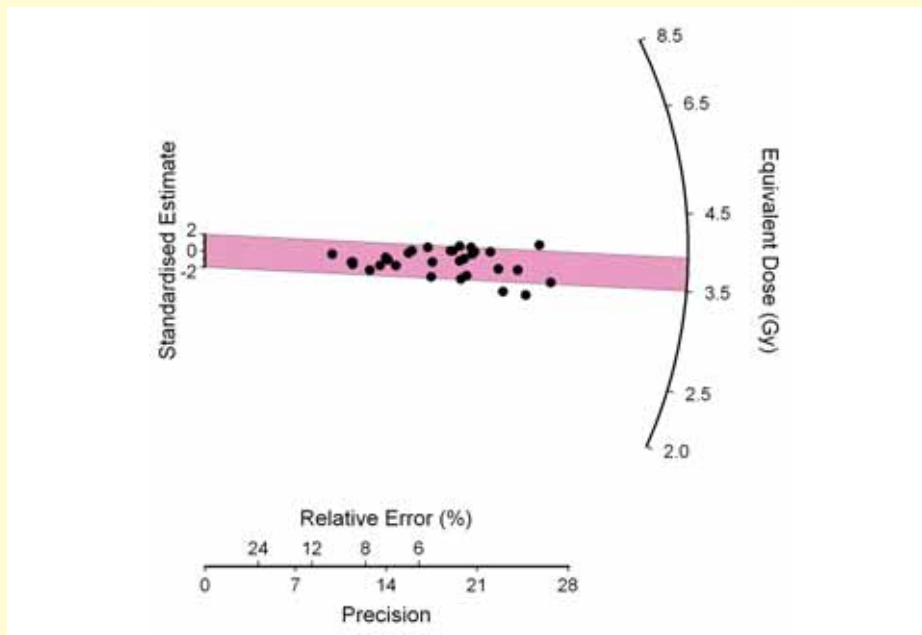


Fig 35 Luminescence data for a quartz aliquot from sample Aber73/BH-1 from Dungeness Foreland: (a) OSL decay curve using blue LEDs (17 mW/cm^2); (b) dose response curve for the same aliquot, giving a D_e value of $3.9 \pm 0.3\text{Gy}$; the regeneration dose at 1.85Gy was repeated and a recycling ratio of 1.06 ± 0.04 calculated, within the acceptable range 0.9 to 1.1.

Fig 36 Radial plot of 31 replicate D_e values for Aber73/BH-1: the majority of the data points lie within the shaded band, indicating that they are consistent with the average D_e within the uncertainties of the individual D_e values. This is the same data as shown in Fig 16a in the form of a histogram.



age was in poor agreement with the other data, and further investigation suggested that this was due to difficulties in calculating the dose rate.

A detailed, three-dimensional view of the evolution of this landscape could be made by combining ages from both an east–west and a north–south transect (Long *et al* 2006; Roberts and Plater 2007). Growth was mostly to the south during the Bronze Age, but more easterly during the post-Roman era, then a switch to more northerly accretion since Saxon times.

To determine the stabilisation of the land surface itself, radiocarbon samples were collected from between gravel ridges. Although a smaller number of radiocarbon samples were measured, they showed that gravel deposition typically occurred within 200–400 years of emplacement of the underlying sand. Together these two dating methods have provided a clear, three-dimensional model of landscape development that provides an important framework for archaeological finds from the area.

The sampling scheme was informed by prior models of the development of this landscape, and thus succeeded in maximising the information from the OSL ages. Taking three samples from each core provided a check on the reliability of the ages against the stratigraphic relationships. Luminescence was ideal for dating the sands, but could not date the gravels. Thus the combination of luminescence dates with radiocarbon dates for the overlying organic sediments gave additional information.

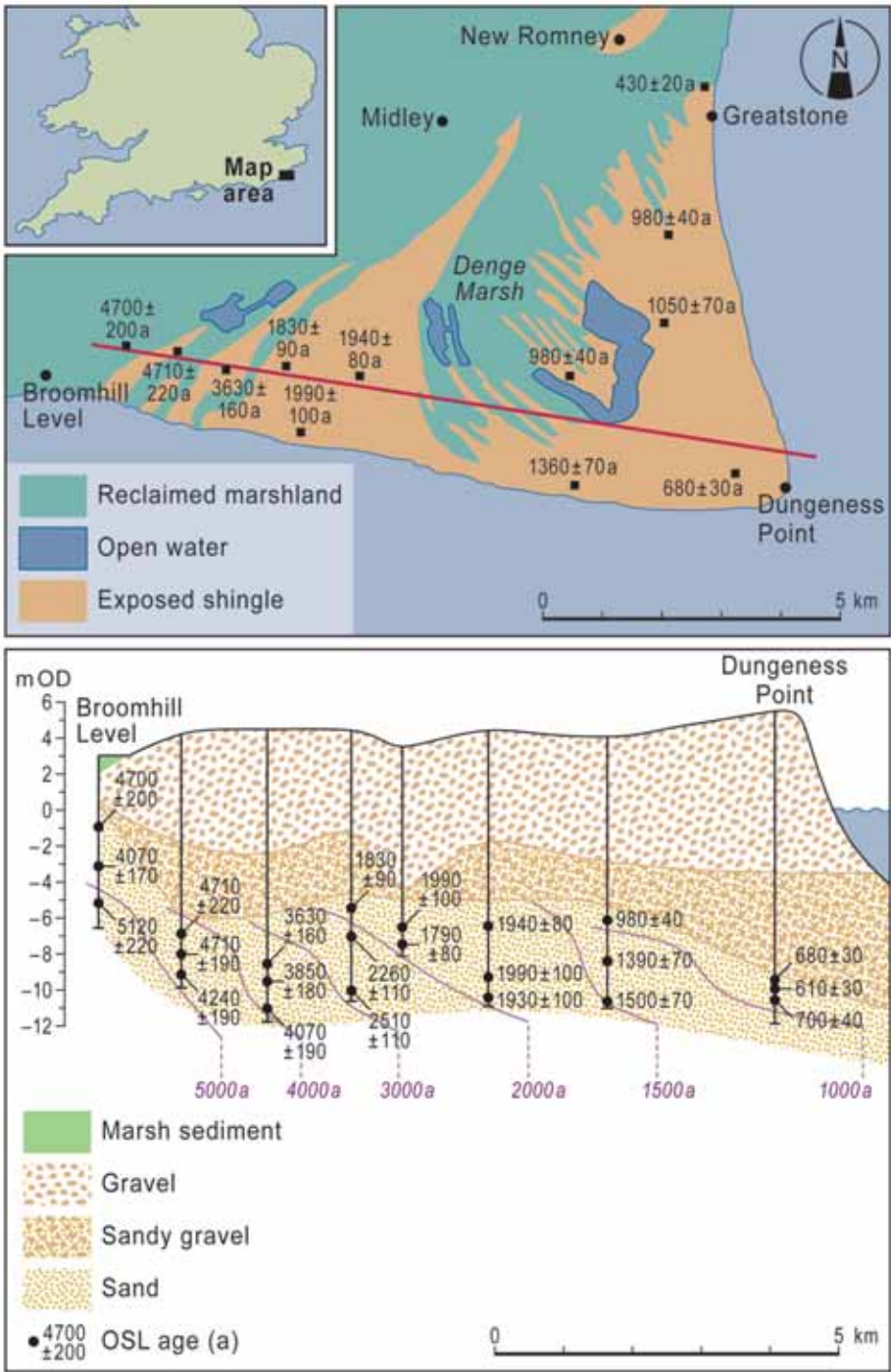


Fig 37 (top) Ages for the uppermost sands from boreholes drilled across Dungeness Foreland. (bottom) A west–east transect (shown as a red line in the upper diagram) across Dungeness Foreland showing OSL ages (in years before AD 2000) for the underlying sands (modified from Roberts and Plater 2007).

Table 5 Part (8 of 37 samples) summary of luminescence results from Dungeness foreland samples (Roberts and Plater 2005; 2007): dose rates adjusted for water contents and calculated for grain size 150–180µm; ages expressed as years before AD 2000, rounded to nearest 10 years (or 10a)

lab code	depth (m)	% water content	equivalent dose (Gy)	no. of aliquots	dose rate (Gy/ka)				age (a)
					beta	gamma	cosmic	total	
73BH-1/1	3.75	21 ± 5	3.70 ± 0.06	31	0.41 ± 0.02	0.25 ± 0.02	0.13 ± 0.01	0.79 ± 0.03	4700 ± 200
73BH-1/2	5.95	24 ± 5	3.11 ± 0.05	33	0.39 ± 0.02	0.28 ± 0.02	0.10 ± 0.01	0.77 ± 0.03	4070 ± 170
73BH-1/3	8.05	25 ± 5	3.44 ± 0.06	18	0.35 ± 0.02	0.25 ± 0.02	0.08 ± 0.01	0.67 ± 0.03	5120 ± 220
73BH-2/1	11.25	25 ± 5	4.31 ± 0.09	18	0.49 ± 0.03	0.37 ± 0.03	0.06 ± 0.01	0.92 ± 0.04	4710 ± 220
73BH-2/2	12.25	25 ± 5	3.81 ± 0.08	17	0.50 ± 0.03	0.37 ± 0.03	0.05 ± 0.01	0.92 ± 0.04	4170 ± 190
73BH-2/3	13.60	25 ± 5	3.55 ± 0.09	12	0.51 ± 0.03	0.29 ± 0.02	0.05 ± 0.01	0.84 ± 0.03	4240 ± 190
73BH-3/1	12.90	25 ± 5	2.37 ± 0.06	17	0.38 ± 0.02	0.22 ± 0.01	0.05 ± 0.01	0.65 ± 0.03	3630 ± 160
73BH-3/2	13.9	25 ± 5	2.42 ± 0.06	18	0.36 ± 0.02	0.23 ± 0.02	0.05 ± 0.01	0.63 ± 0.03	3850 ± 180

13 Fluvial gravels at Broom, Devon

The river terraces of the Axe River, near Broom in Devon, have been known since the 19th century to contain Palaeolithic tools. From 1932 to 1941 a distinguished amateur archaeologist, Charles Bean, amassed more than 900 implements from the gravel quarries actively mining the river terraces (Fig 38). In total more than 1800 handaxes are known from the site (Hosfield and Terry 2001), making it one of the richest Palaeolithic sites in the United Kingdom (Hosfield *et al* in prep).

The sedimentary sequence at Broom can broadly be subdivided into three parts: Lower Gravels, Middle Beds (sand, silt and clay), and Upper Gravels. Based on Bean's records of where artefacts were recovered in the quarry, artefacts apparently occur throughout this sequence. The majority of these handaxes (97%) are made of Upper Greensand chert; most of the remainder are flint. The age of these deposits was uncertain, only being indicated as Acheulian by the handaxe typology and some pollen analyses. The limited archaeological evidence gave no firm indication that the artefacts were in a primary context, but the large proportion (81%) of tools with sharp or very sharp edges suggested that movement had been limited. Thus, although they were within a secondary context, an age for the deposition of the sediments would significantly improve chronological knowledge of the site and its tools. OSL dating was applied to the Broom sediments as part of The Archaeological

Potential of Secondary Contexts project, supported by the Aggregates Levy Sustainability Fund (ALSF). The project illustrates some of the complexities in applying OSL to such old materials.

Excavations since 2000 have yielded few artefacts, so the major aim of the OSL dating was to attempt to construct a chronology for aggradation of the fluvial gravels at the site. While all three parts of the sequence were of interest, the materials most suitable for OSL measurements were sands found in the Upper Gravels and Middle Beds (Fig 38). Researchers from the Geochronology Laboratory, University of Gloucestershire collected nine samples from various outcrops of these units at different locations in the area, using black plastic pipes, or where this was not possible, carved out lithified blocks of material (c 75mm x 75mm x 50mm) and wrapped them in opaque plastic for transport to the laboratory. The outer surfaces of the blocks, which had been exposed to daylight during sampling, were removed in the laboratory, and the resulting sub-sample prepared for OSL measurements. The innermost material was selected for analysis from the samples collected in opaque plastic tubes.

Quartz grains (c 0.1mm diam) were isolated from most of the samples and the SAR procedure used to determine D_e for 12 aliquots of each sample; each aliquot was c 6mg of quartz (4,000 to 10,000 grains). Instead of LEDs, a filtered halogen light system was used to provide optical stimulation (16 mW/cm² at 420–560nm). The OSL signals

obtained were generally bright (Fig 39a); the signal dropped rapidly during measurement, as expected for quartz.

Gamma dose rate measurements were made *in situ* using a gamma spectrometer, complemented by measurements of K, U and Th concentrations by neutron activation analysis (NAA) and ICP-MS in the laboratory. Current water content for each sample (between 7% and 29%) was used for dose rate calculation on the basis that since deposition they would have experienced a wide range of climatic conditions. The dose rate calculated for different samples varied from 1.08Gy/ka to 1.72Gy/ka (Toms *et al* 2005).

The samples analysed from the Upper Gravels and the Middle Beds gave D_e values between c 300 Gy and 550Gy, and exhibited OSL signals showing growth to more than 600Gy (Fig 39b). The natural OSL signals (L_N/T_N) intersected with this dose response curve, yielding finite D_e values, but the natural signals were close to the saturation level of the material (cf Fig 27a).

These results are a good example of the challenges encountered when applying luminescence dating to old samples (see section 5.3). The samples at Broom yielded finite D_e values (and hence ages), but their reliability may be questionable when they are close to saturation. Small variations in experimental uncertainties or systematic effects will have a large impact on the D_e values. For the sample dose response curve



Fig 38 (left) Handaxe from the fluvial gravels at Broom, Devon. (right) Collecting samples at the boundary between the sand-rich Middle Beds and the Upper Gravels for OSL dating (© Dr R Hosfield).



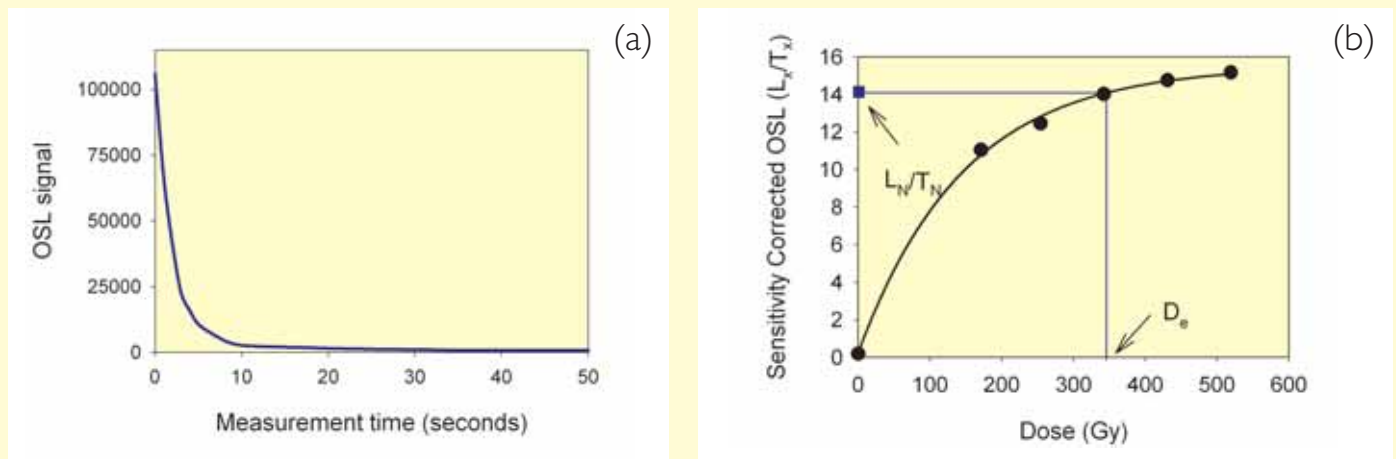
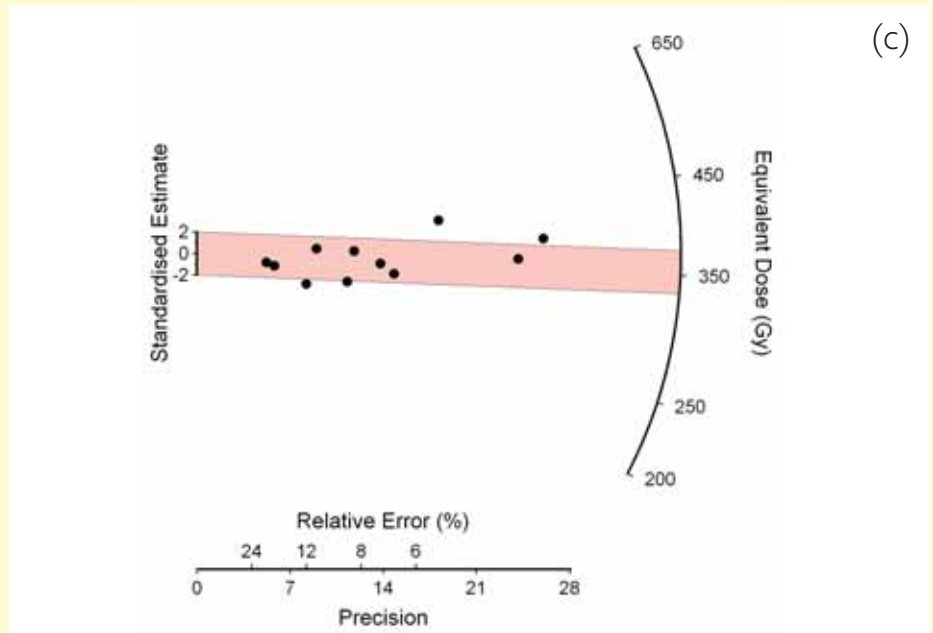


Fig 39 (a) OSL decay curve; (b) a dose response curve for a quartz aliquot from sample GL03008, from the Middle Beds (modified from Toms *et al* 2005); (c) radial plot of eleven replicate D_e measurements for GL03008 – the shaded bar shows the average D_e (353 ± 19 Gy) and points falling within this bar are within 2σ of this value.



shown in Fig 39b, eleven replicate measurements yielded similar D_e values (Fig 39c), but other samples show more variation between aliquots. Such variation can be caused by incomplete bleaching or variations in environmental dose rate between grains; or by variations in the behaviour of grains as they approach saturation. These possibilities are discussed by Toms *et al* (2005). Because a large number of samples had been collected for OSL analysis it was possible to check for consistency between results, and to attempt a Bayesian analysis, which implied ages ranges of 282–324ka for the Middle Beds and 205–292ka for the Upper Gravels (Toms *et al* 2005).

Dating sites such as Broom is important, and OSL provides one of the few techniques applicable. However, interpretation of the ages from this and other sites that lie near the limit of current luminescence dating methods is complex. Publication of the ages from these sites requires the inclusion of detailed supporting luminescence data, so that their validity can be assessed now and in the future.

14 Dating medieval bricks

Only in recent years has the potential of luminescence dating been realised for increasing our knowledge of the phasing of construction of brick-built structures. To evaluate and demonstrate this potential, samples of bricks were collected from several buildings in England whose ages were well known from documentary evidence or dendrochronology: Boston Guildhall, Tattershall Castle, Ayscough Hall, Doddington Hall and Fyde Hall. Their ages range from AD 1390 to AD 1737. Samples were collected by drilling into selected bricks *in situ* to extract solid cores of material (Fig 40). The cores were wrapped in opaque plastic and taken to the laboratory for sub-sampling under laboratory safe-lights.

Measurements of gamma dose rates were made *in situ* using two methods: using a NaI gamma spectrometer and by placing aluminium oxide dosimeters into the drill holes. The gamma dose rate measurements were used as the basis for the gamma dose rate. The beta dose for each sample was measured in the laboratory using a technique known as beta TLD



Fig 40 Using a diamond tipped drill barrel to sample a brick for OSL dating.

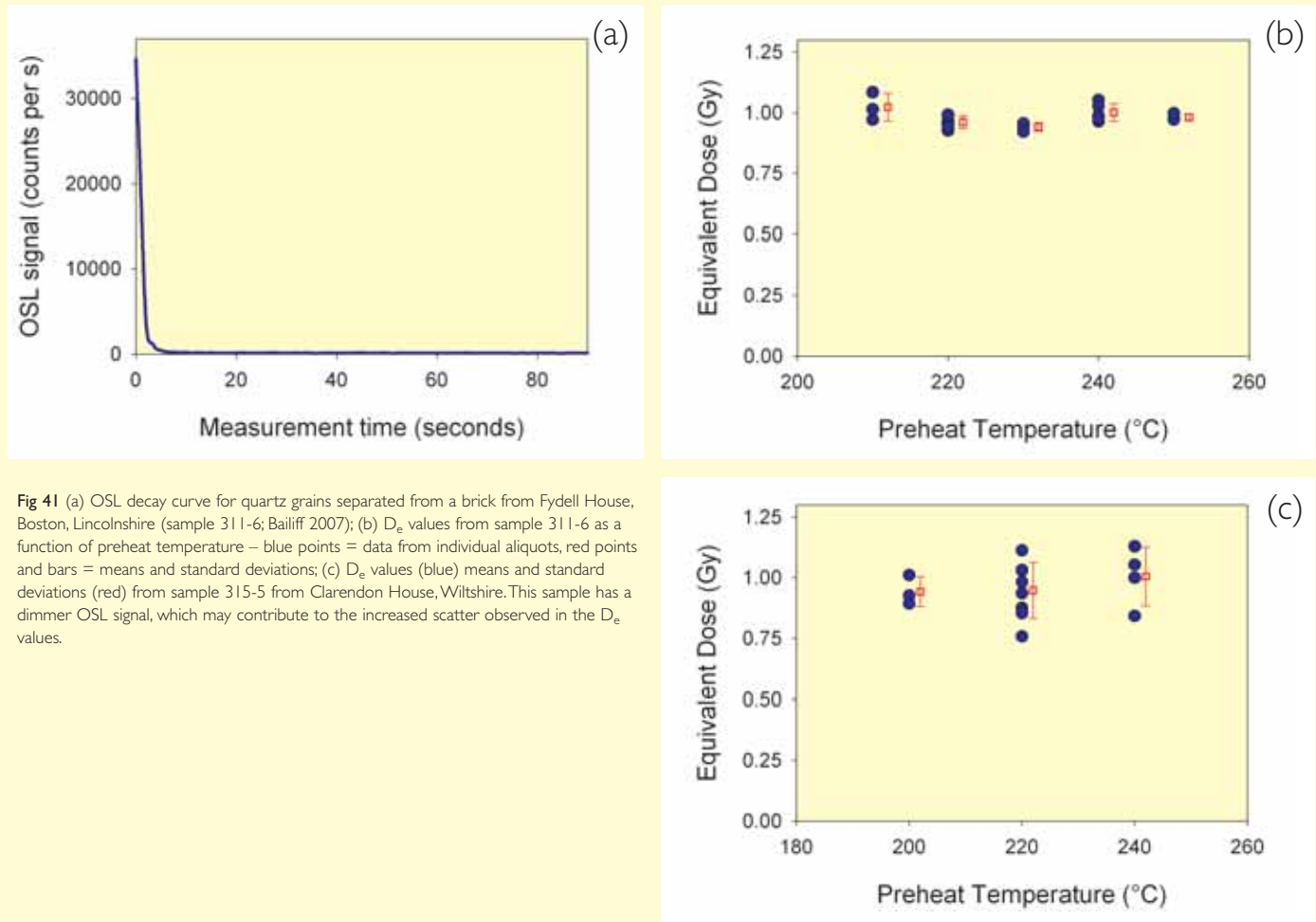


Fig 41 (a) OSL decay curve for quartz grains separated from a brick from Fyde Hall, Boston, Lincolnshire (sample 311-6; Bailiff 2007); (b) D_e values from sample 311-6 as a function of preheat temperature – blue points = data from individual aliquots, red points and bars = means and standard deviations; (c) D_e values (blue) means and standard deviations (red) from sample 315-5 from Clarendon House, Wiltshire. This sample has a dimmer OSL signal, which may contribute to the increased scatter observed in the D_e values.

Table 6 Summary of luminescence data for brick samples from medieval buildings (see Table 7): ages expressed as years before AD 2005; uncertainties include random and systematic sources to one standard deviation.

<i>laboratory</i> (Dur05 OSLq)	<i>preheat</i> <i>temp</i> (°C)	<i>equivalent</i> <i>dose</i> (Gy)	<i>no. of</i> <i>aliquots</i>	<i>beta</i>	<i>dose rate (Gy/ka)</i> <i>gamma</i>	<i>total</i>	<i>age (a)</i>
301-1	220–250	1.27 ±0.02	16	1.89	0.93	2.82 ±0.06	450 ±27
310-1	220–240	2.70 ±0.03	10	2.99	1.38	4.37 ±0.10	617 ±37
311-2	220–250	1.05 ±0.02	11	2.48	1.31	3.79 ±0.08	278 ±17
311-4	210–240	1.15 ±0.04	14	2.58	1.31	3.89 ±0.09	296 ±20
311-6	220–240	0.97 ±0.01	14	1.96	1.26	3.42 ±0.12	284 ±17
315-4	210–230	1.12 ±0.02	6	1.98	1.41	3.39 ±0.07	330 ±18
315-5	200–240	0.95 ±0.03	8	2.11	1.35	3.46 ±0.08	273 ±18
317-1a	200–240	1.36 ±0.01	8	2.05	1.19	3.24 ±0.07	419 ±24
317-1b	210–230	1.56 ±0.04	9	2.42	1.21	3.64 ±0.08	427 ±27
318-1	200–240	1.79 ±0.02	11	2.09	1.16	3.25 ±0.07	550 ±33
318-2	200–240	1.84 ±0.03	8	2.26	1.07	3.33 ±0.08	552 ±34
319-1	200–240	1.93 ±0.02	7	2.14	1.32	3.46 ±0.08	558 ±32

Table 7 Comparison of luminescence ages from OSL measurements of quartz from bricks in medieval buildings: luminescence ages from Table 6 given as years before AD 2005 and as calendar dates for comparison with independent assessment of the ages from archival and other sources (see Bailiff 2007 for details)

<i>lab code</i> (Dur05 OSLq)	<i>building</i>	<i>luminescence age (a)</i>	<i>calendar date</i>	<i>independent assessment</i> <i>or assigned date range</i>
301-1	Alford Manor House, Alford, Lincs	450 ±27	1555 ±27	1611–1615
310-1	St Mary's Guildhall, Boston, Lincs	617 ±37	1388 ±37	1390–1395
311-2	Fyde House, Boston, Lincs	278 ±17	1727 ±17	1700–1726
311-4	Fyde House, Boston, Lincs	296 ±20	1709 ±20	1700–1726
311-6	Fyde House, Boston, Lincs	284 ±17	1721 ±17	1724–1726
315-4	Clarendon House, Wiltshire	330 ±18	1688 ±18	1667–1690
315-5	Clarendon House, Wiltshire	273 ±18	1730 ±18	1717–1737
317-1a	Doddington Hall, Doddington, Lincs	419 ±24	1586 ±24	1593–1600
317-1b	Doddington Hall, Doddington, Lincs	427 ±27	1576 ±27	1593–1600
318-1	Tattershall Castle, Tattershall, Lincs	550 ±33	1455 ±33	1445–1450
318-2	Tattershall Castle, Tattershall, Lincs	552 ±34	1453 ±34	1445–1450
319-1	Ayscoughfee Hall, Spalding, Lincs	558 ±32	1447 ±32	1450–1455

(thermoluminescence dosimetry), which uses a sensitive luminescence phosphor to measure the beta activity of the sample directly. The advantage for this study is that the beta dose will originate from entirely within the specific brick being sampled. For these samples the beta dose contributes c 64% of the total dose (Table 6).

Further emission counting methods were used to measure the alpha activity (normally assumed to be zero) of the quartz grains used for luminescence measurements. The measurements here suggested that the alpha activity was c 1% of the total dose rate (between 2.8 Gy/ka and 4.4 Gy/ka). However, a small allowance was made for this by adding 0.03 ±0.02 Gy/ka to the dose rate calculated for each sample.

The cosmic dose rate was calculated using the position of the bricks within each structure as a guide to their likely exposure. The water content of the bricks was less than 2% for all samples except one, whose value was 3–4%. For calculation of the average dose rate, a water content of 3 ±1% was used for all samples except for the one whose value was higher; for that sample 5 ±1% was used.

Coarse grains of quartz (c 0.1 mm diam) extracted from the bricks were used for OSL measurements. OSL signals from aliquots containing 1–2 mg of the quartz was measured using optical stimulation provided from either blue LEDs (c 50 mW/cm²) or a filtered halogen light (c 30 mW/cm²). The brightness of the OSL signal varied substantially between the

different samples, and the dimmest sample had a signal to background ratio close to 1.0. These OSL signals were characterised by a rapid drop at the beginning of the measurement, as expected for quartz (Fig 41a). Aliquots that had large statistical fluctuations in their OSL signals after background subtraction were excluded from further analysis.

The samples were measured using a SAR procedure, giving between 6 and 16 D_e values for each brick that could be combined for use in the OSL age calculation (Table 6). The selection of an appropriate preheat temperature was done by measuring D_e at a variety of preheats for each sample. For some samples the D_e values were reproducible, while more scatter was observed for others

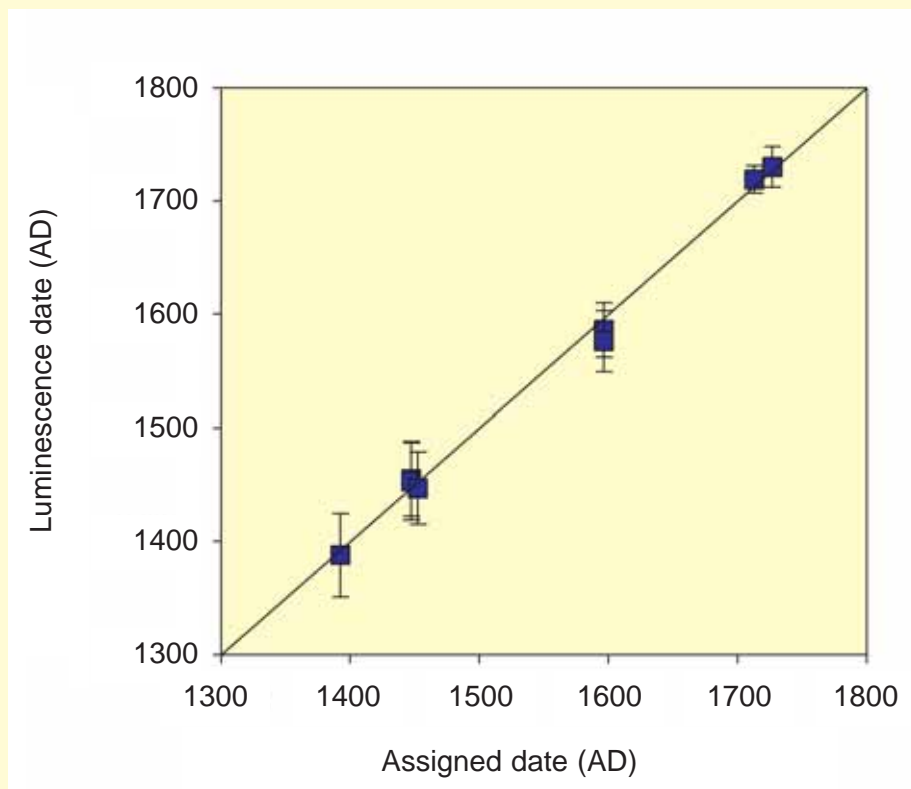


Fig 42 Comparison of ages obtained using OSL dating of bricks and dates assigned to parts of the built structures based on documentary and dendrochronological evidence (Bailiff 2007).

(Fig 41b and c).

The comparison between the OSL ages and the assigned dates was extremely good (Table 7 and Fig 42), and the typical error on the OSL ages was only 25 years, even allowing for systematic errors. Further analysis of bricks from another site, Alford Manor (301-I), gave OSL dates some 55 years earlier than the assigned ages, and subsequent structural analysis showed that the part of the building that had been sampled had been rebuilt, possibly re-using bricks from an older structure.

Although the use of OSL for dating bricks has not been common in England, the potential of the method is clear; both for dating structures whose age is unknown, and potentially for detecting areas that have been rebuilt, or



Summary

Luminescence is the light emitted by minerals after they have been exposed to radioactivity. It provides a method for dating materials that were either heated in the past (pottery, burnt flint and bricks) or that were exposed to daylight (geological sediments). The event being dated is the last time the sample was heated or exposed to daylight. The method is applied to the mineral grains within a sample, and the majority of analyses are undertaken on quartz. The method is based on the observation that exposure of a sample to radioactivity increases its luminescence signal and provides the basis for the chronometer. Radioactivity is ubiquitous in the natural environment, originating from uranium, thorium and potassium surrounding the sample, and from cosmic rays that originate from beyond the Earth.

Luminescence measurements are used to calculate the total radiation dose to which the sample has been exposed since the event being dated. This quantity is known as the equivalent dose (D_e), and is measured in the units Gray (Gy).

The rate at which a sample is exposed to radioactivity in its natural environment can be measured either by chemical methods or by directly measuring the emission of radioactivity. This is termed the dose rate and has the units Gray per year (Gy/year). The age of the sample is calculated by dividing the equivalent dose by the dose rate.

Two types of luminescence measurement can be made in the laboratory: heating the sample results in thermoluminescence (TL); or stimulating the sample using light of a limited wavelength, which results in the emission of Optically Stimulated Luminescence (OSL). TL is used to date materials when the event being dated is the last time they were heated. OSL is the appropriate technique when dating the last exposure to daylight. OSL can also be used for heated materials.

Unlike radiocarbon ages, luminescence ages do not require calibration. The unit annum (abbreviated to 'a') should be used instead of 'years', or ka (thousands of years) where appropriate. The term BP (before present) should never be used for a luminescence age; it should only be used for radiocarbon ages. The age range over which luminescence dating works varies from sample to sample depending upon the nature of the quartz and the dose rate specific to that sample. In ideal circumstances the method works on samples from as young as a few decades to as old as several hundred thousand years.

The precision of luminescence ages is typically 5–10%; thus ages of $50 \pm 5a$, $5,000 \pm 500a$ and $50 \pm 5ka$ all have uncertainties of 10%. The largest single source of uncertainty is the water content of the samples, which affects the dose rate.

Calculating luminescence ages involves measurement of many parameters, and reports from luminescence laboratories should include all of this data and be included in the project archive. When reporting dates in monographs or academic papers it is essential to incorporate sufficient data to enable readers to judge the quality of the age. Particular problems occur for very young samples (< 1000 years) where low luminescence signal levels may degrade the precision, and for very old samples ($> 50,000$ years) where saturation of the luminescence signal limits the application of the method and introduces larger errors as the sensitivity of the technique is reduced. When dating the last exposure of materials to daylight additional complications can arise if the sample was not exposed to daylight, for a sufficient period of time to completely remove any pre-existing signal.

Replicate measurements of D_e from a single sample can be used to test whether this occurred, by looking to see whether the D_e values are consistent from one sub-sample to another. Statistical methods are available designed to calculate the age in situations where the signal was not completely removed at deposition.

A minimum requirement when reporting dates is to include diagrams illustrating the luminescence signal measured and the growth of the luminescence signal with radiation dose. Where there are concerns about whether the sample was exposed to sufficient daylight at deposition to reset the signal the results of replicate measurements of D_e should be shown. A table of the results should also be included and at a minimum this should list the laboratory code, the material analysed, dose rate, D_e , the number of replicate measurements used to calculate D_e and the calculated age. Luminescence ages are calculated in years before the date of measurement, and the date of measurement needs to be stated. The text associated with the table and the figures should also include a brief description of the analytical methods used for D_e and dose rate measurements, and a description of how the water content was assessed.

Given the complex nature of luminescence dating it is generally recommended that

publication is undertaken jointly with the luminescence laboratory involved with the measurements so that they can provide expert input into the presentation of the data.

In text, luminescence ages should be quoted with their unique laboratory code, and the date listed with its associated uncertainty. Ages may be expressed in years before some datum (eg AD 2007), or as calendar dates BC or AD.

Luminescence provides a powerful technique that complements other dating methods. Given the complexity of the method it is recommended that the luminescence laboratory be consulted early in the project planning stage to provide advice about the optimal sampling conditions and to assist in designing a sampling strategy.

Further reading

The fundamentals of luminescence dating were spelt out in detail by Aitken (1985), and a brief version was included in his later book (Aitken 1990). Subsequently Roberts (1997) provided a comprehensive review of the use of luminescence in archaeological applications worldwide. While these sources remain excellent for discussions relating to the dose rate and the use of thermoluminescence, they predate the major developments in the use of the optically stimulated luminescence (OSL) signal from quartz, and the SAR procedure now used. More recent reviews by Lian and Roberts (2006) and Duller (2004) focus on developments in the use of OSL for estimation of D_e , and Jacobs and Roberts (2007) provide an overview of the current state-of-the-art in applying single-grain methods. Walker (2005) gives a broader summary of the use of luminescence in Quaternary sciences. Wintle (2008) looks at the history of the development of luminescence dating with particular attention to archaeological

applications.

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Glossary

aliquot a sub-sample of the material whose luminescence signal is being measured. Typically, aliquots of sand-sized grains are 1–5mg of the sample. For fine-grain (0.004–0.011mm diam) samples an aliquot will weigh 1mg or less. The grains of quartz, feldspar or other mineral being measured are mounted on a steel or aluminium disc c 9.8mm diameter.

alpha particle one type of radiation emitted from atoms when they undergo radioactive decay; an alpha particle is a helium nucleus and consists of two neutrons and two protons that can travel up to 20µm.

anomalous fading a luminescence signal that is anomalously unstable compared with theoretical predictions. This affects feldspars, and if unaccounted for would lead to luminescence ages that are too young.

beta particle one type of radiation emitted from atoms when they undergo radioactive decay; a beta particle is an electron. They can travel up to 2mm.

bleaching exposing a sample either to daylight or to an artificial light in order to remove the trapped electron population.

conduction band a conceptual part of a crystal where electrons can move freely from one part of the crystal to another. In most minerals electrons need additional energy (eg from heating or exposure to light) to reach the conduction band, and electrons can only exist in the conduction band for a limited period of time.

cosmic rays a type of high energy, penetrating radiation made up of charged particles that originate beyond the Earth; can be deflected by the Earth's magnetic field.

dose rate (also known as the effective dose rate) the total radiation dose to which a sample has been exposed in a given period of time; normally expressed as the radiation dose per year (Gy/a) or per thousand years (Gy/ka).

dose response curve the increase in a luminescence signal as a function of the radiation dose it has absorbed before measurement.

equivalent dose the laboratory estimate of the radiation dose that the sample received during burial; commonly, and preferably, abbreviated D_e ; other terms sometimes used include

palaeodose (P).

gamma ray one type of radiation emitted from atoms when they undergo radioactive decay; a type of electromagnetic radiation that can travel up to 300mm.

glow curve the **thermoluminescence** signal from a sample as it is heated from room temperature to typical temperatures of c 400°C or 500°C.

Gray (Gy) the SI unit used for absorbed radiation dose: one Gray is equivalent to one joule of energy being deposited in each kilogramme of a sample.

growth curve see 'dose response curve' (the preferred term).

incomplete bleaching term applied to sediments that did not receive sufficient exposure to daylight before deposition to reset the trapped electron population, making it more complex to date, and requiring many replicate D_e measurements and use of statistical methods.

infrared stimulated luminescence (IRSL) the luminescence signal emitted when a mineral is exposed to infrared radiation (typically c 880nm, beyond the visible range); used for dating feldspars.

isotope one form of an element: different isotopes of an element have the same chemical properties, but different atomic masses due to different numbers of neutrons (eg ^{12}C , ^{13}C and ^{14}C , or ^{235}U and ^{238}U , or ^{39}K and ^{40}K – the number beside the element denotes the atom's mass). Thus, an atom of ^{12}C has an atomic mass of 12, while ^{14}C is heavier with a mass of 14. Unstable isotopes – such as ^{14}C , ^{235}U , ^{238}U and ^{40}K – emit radiation as they transform into a different isotope or element.

luminescence a phenomenon exhibited by many naturally occurring materials and used as the basis for dating. Luminescence is light emitted by some minerals when thermally or optically stimulated following exposure to ionizing radiation. The light is normally weak, invisible to the naked eye, but detectable in the laboratory using a photomultiplier tube.

optical dating dating using the OSL signal.

optically stimulated luminescence (OSL) the light emitted from a sample when it is stimulated by exposure to light.

photomultiplier tube (PMT) a highly sensitive device for measuring very small amounts of

light (eg luminescence).

photoluminescence (PL) another term for optically stimulated luminescence.

preheat (cutheat) heating a sample prior to measuring its luminescence in order to remove unstable signals; samples are typically heated in the range 150°C to 300°C.

radial plot a type of graph commonly used to display multiple D_e estimates, determined for a single sample; each D_e value is shown as a separate point together with the uncertainty associated with that measurement, enabling visual differentiation between those points known precisely and those known less well.

radioactivity the spontaneous disintegration of atoms by emission of matter and energy, including **alpha** and **beta particles**, and **gamma rays**.

recycling ratio or **recycling test** one of a range of tests commonly made as part of a SAR sequence. Recycling tests involve applying the same regeneration dose in two different cycles, and giving the ratio of the sensitivity corrected luminescence signals from the two measurements – recycling ratio. If the SAR protocol and the sensitivity correction are working appropriately, then the recycling ratio should be 1. Values from 0.9 to 1.1 are considered acceptable; values outside these limits indicate that the SAR protocol is not working as expected and that the data for that aliquot should be discarded.

single aliquot regenerative dose (SAR) protocol a sequence of laboratory operations commonly used to measure D_e ; normally used on quartz, a main advantage of which is that by using a test dose, any changes in the luminescence sensitivity of the sample are explicitly monitored and corrected.

test dose a radiation dose, given to an aliquot in the laboratory, and used to monitor whether the aliquot is undergoing changes in its luminescence properties during the sequence of measurements that make up a SAR protocol.

thermal transfer the movement of electrons from one trap to another in a crystal by heating; may cause problems when dating very young sediments, so a preheat test should be used to determine the preheat temperature to minimize the problem.

thermoluminescence (TL) the light emitted when a crystalline material, which has previously been exposed to radioactivity, is

heated. It results from the release of energy stored within the crystal and is different from the incandescence (black body radiation) that is observed at higher temperatures (especially $> 500^{\circ}\text{C}$). Incandescence is observed by heating the sample a second time, and is subtracted from the first TL measurement.

trap or **trapping centre** a site in a crystal where electrons can become lodged and remain stored for some period of time (varying from fractions of a second to millions of years depending on the nature of the trap). Some traps are formed by chemical impurities in the crystal; others may relate to structural defects. Stimulating the crystal by heating it or exposing it to light can eject electrons from

traps into the conduction band.

Appendix I Sources of advice on Scientific Dating from English Heritage

Within English Heritage the first point of contact for general archaeological science enquiries, including those relating to luminescence dating, should be the English Heritage regional science advisors, who can provide independent, non-commercial advice. Such advisors are based either in universities or in the English Heritage regional offices. Please contact regional advisors currently based in universities at their university address.

North West

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Vacant
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South East

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fax: 020 7383 2572
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Specific advice on scientific dating, including luminescence, can be sought from the English Heritage Scientific Dating Team (Alex Bayliss, John Meadows, and Isabelle Parsons).

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English Heritage
1 Waterhouse Square, 138–142 Holborn
London EC1N 2ST
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fax: 020 7973 3001
e-mail: Alex.Bayliss@english-heritage.org.uk

Appendix 2 Luminescence laboratory contact details

An essential part of the successful application of luminescence dating is early discussion between the field project director and the specialist undertaking the analysis. Contact details of laboratories in the United Kingdom that are equipped to undertake luminescence dating are given below. Note that not all laboratories undertake all forms of analysis nor do they all provide commercial services. The laboratories are listed in alphabetical order based upon their location or commercial name. Details were correct at the time of writing.

Aberdeen

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Aberystwyth

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Institute of Geography and Earth Sciences
Aberystwyth University
Ceredigion SY23 3DB
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website:
www.aber.ac.uk/quaternary/luminescence

Cheltenham

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Loughborough

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fax: 0115 9515249
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Oxford

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Scottish Universities

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St Andrews

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TL Quaternary Surveys

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Front cover: Collecting luminescence samples under black plastic, Ripon, Yorks.

Micrographs from top to bottom:

- i. Measuring dose rate in Silbury Hill
- ii. Quartz grains and their luminescence signals
- iii. Laser beam for stimulating luminescence from single grains

Back cover: Luminescence sampling, Broom, Devon



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